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AFML-TR-66-75
PART III

NEW ABLATIVE PLASTICS AND COMPOSITES, THEIR FORMULATION AND PROCESSING

B. G. KIMMEL AND G. SCHWARTZ
Hughes Aircraft Company

TECHNICAL REPORT AFML-TR-66-75, PART III

JUNE 1968

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AFML-TR-66-75
Part III

Report No. P68-78
HAC Ref. No. A6230

NEW ABLATIVE PLASTICS AND COMPOSITES, THEIR FORMULATION AND PROCESSING

June 1968

B. G. KIMMEL AND G. SCHWARTZ
Hughes Aircraft Company

FOREWORD

This report was prepared by Hughes Aircraft Company, Culver City, California, under USAF Contract No. AF 33(615)-2418. This contract was initiated under project No. 7340, "Non-Metallic and Composite Materials," Task No. 734001, "Thermally Protective Plastics and Composites." The work was administered under the direction of the Nonmetallic Materials Division, Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. P. F. Pirrung (MANC) acted as project engineer.

This report covers work from February 1967 to February 1968. Work accomplished from February 1965 to February 1966 was reported in AFML TR 66-75. Work accomplished from February 1966 to February 1967 was reported in AFML TR 66-75, Part II.

Previous work on this program was performed under USAF Contract No. AF 33(657)-8621 and will be found in ASD TR 63-568, Part I, ML TDR 64-222 and AFML TR 65-94.

Report was submitted by the authors April 1968.

This technical report has been reviewed and is approved.

R. G. Spain

R. G. SPAIN, Acting Chief
Plastics and Composites Branch
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ABSTRACT

Precise processing techniques were used in preparing new ablative plastics and composites. This research involved the use of novel heat-resistant resins such as:

- bisbenzimidazobenzophenanthroline
- branched, crosslinked polyphenylenes
- chrome based metal organic phenolic
- PBI-carborane
- phenyl aldehyde
- poly(α, α'-diphenyl-xylylidine)
- polyaminoborane
- polyimidazopyrrolone
- polyimide
- polyphenylene sulfide
- poly(perfluorophenylene)
- tungsten based metal organic phenolic

Novel reinforcements included:

- boron nitride fibers
- high modulus carbon yarn
- high modulus graphite fabric and yarn
- silicon carbide whiskers

Resin impregnation techniques used in preparing research specimens included spatula or brush coating, dip coating, soaking, and dry powder layup.

The following research specimens of controlled composition were prepared and submitted to the Air Force Materials Laboratory for hyperthermal evaluation:

- pellet specimens, 3/4-inch diameter by 1/2-inch long
- rocket nozzle assemblies
- cylinders, 1 inch diameter by 2 inches long
- laminate, 7 x 7 x 1/4 inch
- laminated squares, 2 x 2 x 1/2 inch

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SECTION I
INTRODUCTION

New polymeric materials and reinforcements have been developed in Government and industry research programs. Many of these materials offer considerable promise for use in high-performance ablative plastics.

The program objectives are to select promising ablative materials for further study and develop suitable fabrication procedures for preparing small ablative composites containing these new materials.

The principal work during this 12-month period of the program was the continued use of precise processing techniques in fabricating research specimens of closely controlled composition. Specimens were produced consistently with a resin content within a ±2-percent range. In all experiments, all pertinent processing information and data were recorded to allow later duplication of any test specimens required for further tests. These processing data can be used in scaling up the processes if required. Specimens prepared under this contract have been forwarded to the Nonmetallic Materials Division, Air Force Materials Laboratory, for subsequent hyperthermal evaluation.

During this period, newly developed resins and reinforcements, which are becoming available in research quantities, were used to fabricate ablative composite specimens. These specimens will be subsequently characterized for possible use in high speed entry and rocket exhaust environments. Materials intended for potential entry environments will be characterized with an air arc heater. This research is being performed under AF 33(615)3923 with the Avco Corporation, SSD. Rocket nozzle specimens will be characterized using a liquid propellant motor or a solid propellant motor simulator under Contract AF 33(615)-1632 with the Aeronutronic Division, Philco Corporation.

SECTION II
SUMMARY

Precise formulation and processing techniques were used in the preparation of ablative composites of controlled composition containing new polymeric materials and reinforcements.

Formulating, molding, and postcuring conditions were varied, as required, to produce test specimens of high quality from a wide range of resins and reinforcements. New resins investigated included:

- bisbenzimidazobenzophenanthroline
- branched, crosslinked polyphenylenes
- chrome based metal organic phenolic
- PBI-carborane
- phenyl aldehyde
- poly(α, α'-diphenyl-xylylidine)
- polyaminoborane
- polyimidazopyrrolone
- polyimide
- polyphenylene sulfide
- poly(perfluorophenylene)
- tungsten based metal organic phenolic

Novel reinforcements included:

- nitride fibers
- high modulus carbon yarn
- high modulus graphite fabric and yarn
- silicon carbide whiskers

In addition, a large quantity of research specimens was fabricated using standard resins such as phenolics or epoxy novolacs and standard reinforcements such as Refrasil, carbon cloth, or graphite cloth. In many cases, a standard reinforcement was combined with a new resin while a new reinforcement was combined with one of the standard resins.

During the period covered by this report, the following specimens were prepared and shipped to Air Force Materials Laboratory:

4 cylinders
1 laminate
3 laminated squares
132 pellet specimens
39 ASD No. 4 rocket nozzle assemblies
6 thermogravimetric analysis specimens

SECTION III

GENERAL SPECIMEN PREPARATION PROCEDURES

1. GENERAL DISCUSSION

Precise formulation and processing techniques were developed and applied in the fabrication of ablative composites containing new polymeric materials and reinforcements. Five main types of test specimens were prepared and submitted under this program:

- Cylinders; 1.000 ±0.001 inch in diameter x 2.000 inches long
- Laminates; 7 x 7 x 1/4 inch
- Laminated squares; 2.000 x 2.000 x 0.502 ±0.002 inch
- Pellet specimens; 0.750 inch in diameter x 0.502 ±0.002 inch long
- Rocket nozzle assemblies; ASD No. 4

A complete description of all test specimens fabricated and delivered during the period covered by this report is given in Tables I through X in the Appendix.

Tables I, II, and III give density, Barcol hardness, composition, and a brief description of cylinders, one laminate, laminated squares, pellet specimens, and rocket nozzles.

Table IV lists all specimens by test specimen data sheet number. It also gives the specimen type, material code, and other information such as the date requested and shipped.

Table V lists all test specimens according to type of reinforcement. Table VI lists all test specimens according to type of resin.

Tables VII, VIII, and IX give the fabrication details for cylinders, a laminate, laminated squares, pellet specimens, and nozzles.

Table X lists material sources for resins, reinforcements, and fillers used.

Table XI is a cumulative index listing all specimens shipped under Air Force Contracts AF 33(657)-8621 and AF 33(615)-2418, by data sheet number, type of specimen, material code, and AFML Technical Report numbers. Table XII lists the material symbol codes used.

The composition of the test specimens was maintained in almost all cases within the range of ±2 weight-percent of the required nominal composition. This was done by carefully controlling each step of the fabrication process from the initial coating of the reinforcement to the final postcure of the molded or laminated composite. Past experience was used in making allowance for the weight loss (change in composition) of the coated reinforcement which takes place upon drying, B-staging, curing, and postcuring.

All reinforcements except glass and high silica content cloth were oven-dried two to three hours at 240°F prior to coating with resin. All subsequent calculations were based on this dry reinforcement weight. Carbon and graphite cloth have been found to lose as much as 10 weight-percent on drying.

2. TYPES OF IMPREGNATION

Several methods of impregnating the reinforcements were used:

- Spatula or brush coating
- Dip coating
- Soaking
- Dry powder layup

A. Spatula or Brush Coating

This method of impregnation is used only on cloth. Fabric is cut to a size sufficient to allow the blanking or cutting out of the proper number of plies for the molding (Figure 1). The dry cloth is weighed and laid out on a piece of cellophane. The proper amount of resin is weighed out and thinned, if necessary, to coating consistency. The resin is poured over the fabric and uniformly distributed over the cloth with either a spatula or a 1-inch wide paint brush. The impregnated material is dried on the cellophane for 15 to 20 minutes, then hung up to dry for about 1 hour at room temperature. After drying at 160°F for 20 to 60 minutes, the cloth is weighed and the resin content calculated. Excess resin is removed by wiping the surface with a paper tissue soaked in thinner. However, if additional resin is needed, it is added to the back side of the cloth and uniformly distributed by either spatula or brush. When the desired resin content is reached, the fabric is B-staged to form a prepreg. The final resin content is then calculated from the final coated weight.

B. Dip Coating

This method of impregnation is used only on cloth. A weighed piece of dry cloth is repeatedly passed through a small dipping tray until the required amount of resin is obtained. When a large number of dips are required, the cloth is allowed to dry after every fourth dip. The drying time, from 5 to 30 minutes, depends on the resin system. Dip coating is usually used in place of spatula coating under the following circumstances:

- With solutions containing small percentages of resin solids

Figure 1. Blanking of Prepreg in Punch Press
(HAC Photo 4R01228)

- With viscous resin solutions with large amounts of thinner added to obtain satisfactory coating properties.
- When coating carbon or graphite cloth with a solution containing a high percentage of resin solids. These types of cloth tend to powder when spatula coated with a solution with a high solids content

When the correct resin content is obtained, the cloth is B-staged and the final resin content is calculated from the weight of the B-staged cloth.

C. Soaking

This method is used with yarns, filaments, or fibers which wet readily. The dry material is placed in a beaker and thinned resin solution containing a weighed amount of resin solids is added. The reinforcement is allowed to soak for 60 minutes in air before the excess solvent is removed by evaporation under vacuum. After drying in an oven at 160°F for 60 minutes, the resin content of impregnated reinforcement is calculated from the increase in weight.

The resin content of the prepreg is increased or decreased when required, by pouring additional resin or solvent over the material and filtering off the excess. The material is B-staged after obtaining the proper resin content.

D. Dry Powder Layup

Cloth cannot be impregnated when the resin used is a dry insoluble powder. Specimens are prepared by sprinkling resin between plies of reinforcement. Pieces of cloth are blanked into plies and when required are dried in an oven at 240°F for two hours. A calculated amount of resin is sprinkled between plies with each addition of resin and cloth being weighed on an analytical balance. The resin and reinforcement are weighed into a preform holder and transferred into the cold mold prior to molding.

3. MOLDING AND POSTCURING

The moldings and laminates required for this program were made in precision laboratory presses under closely controlled conditions of temperature, pressure, contact time, and cure time.

Oven postcures were accomplished in mechanical convection ovens equipped with cam-type programming controllers. Inert atmosphere postcures were conducted with the moldings or laminates enclosed in a stainless steel fixture through which a slow stream of argon was passed (see Figure 2).

Figure 2. Specimens Being Prepared for Postcure in Argon Atmosphere (HAC Photo 5R01227)

SECTION IV
FABRICATION PROCEDURES

Fabrication procedures for each type of specimen are given in the following paragraphs.

1. CYLINDERS

Fabrication procedures for different compositions of 1-inch diameter x 2-inch long cylindrical specimens are listed in Section V, Specific Specimen Procedures. Molding conditions are listed in Table IX.

2. LAMINATES (1/4 INCH OR LESS IN THICKNESS)

One laminate, 7 x 7 x 1/4 inch, was prepared as follows. B-staged prepreg was cut to the proper size, randomized, stacked, and wrapped in cellophane. The resulting layup was placed between 1/8-inch aluminum cauls and loaded into the press. The laminate was cured using the molding conditions listed in Table IX. After postcure, the final resin content was determined. The laminate was then trimmed and squared by sawing with a diamond bandsaw. Finally, the density was calculated from the dimensions and weight of the specimen.

3. LAMINATED SQUARES

Laminated squares are machined from laminates at least 5/8-inch thick. The prepreg for molding the laminate is prepared by spatula, brush, or dip coating. After B-staging, the material is cut into plies sufficiently large to allow the machining of the required number of specimens. The prepreg is laminated in an open laminating fixture (Figure 3) which allows the escape of excess volatiles and prevents slippage of plies during cure. Molding conditions are listed in Table IX. Specimens were rough cut prior to postcure to minimize the possibility of "blow-up." Diamond tools were used to machine postcured pieces to final dimensions.

4. PELLET SPECIMENS

When sufficient material is available, pellet specimens are machined from cylindrical moldings either 2 inches or 3-1/2 inches in diameter. The parts are usually molded at least 5/8-inch thick to ensure sufficient material for machining. Individual pellets are molded when not enough material is available for molding the larger discs. Pellet specimens machined from the larger discs would be expected to vary only slightly in composition compared with specimens individually molded. Before molding a large disc, a 3/4-inch diameter pellet is

Figure 3. Prepreg Being Molded in Laminating Fixture (HAC Photo R108700)

made to determine the molding characteristics of the prepreg. The charge weight for the large disc is then calculated using the following formula:

$$\text{Charge weight of large disc} = \frac{\begin{pmatrix}\text{Diameter of}\\ \text{large disc}\end{pmatrix}^2 \begin{pmatrix}\text{Desired thickness}\\ \text{of large disc}\end{pmatrix}\begin{pmatrix}\text{Weight of}\\ \text{3/4-inch disc}\end{pmatrix}}{\begin{pmatrix}\text{Diameter of}\\ \text{3/4-inch disc}\end{pmatrix}^2 \begin{pmatrix}\text{Thickness of}\\ \text{3/4-inch disc}\end{pmatrix}}$$

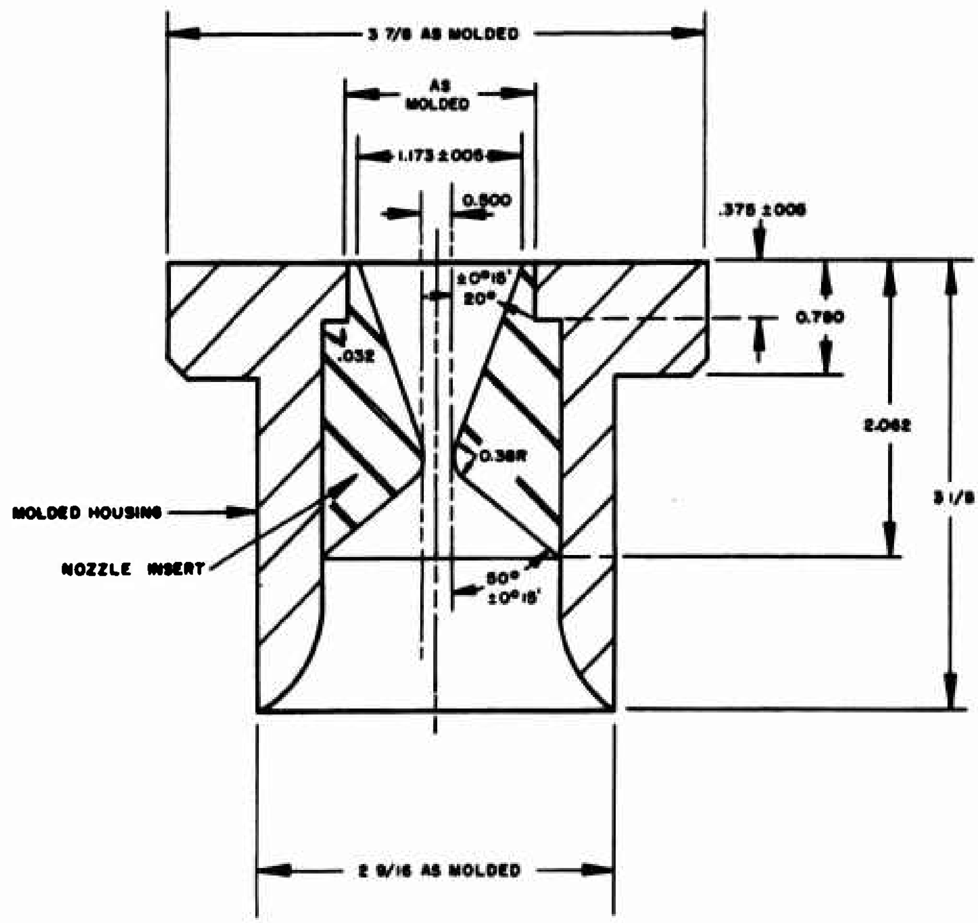
The number of plies needed to mold a laminated disc is calculated by first averaging the weights of five plies. The charge weight is then divided by the average weight per ply for the answer.

Discs were molded and postcured using the conditions listed in Table VII. The density is determined from the dimensions and weight. Pellet specimens are cut from large discs using a diamond bandsaw and all specimens are machined to final dimensions using a Carboloy cutter.

5. ROCKET NOZZLE SPECIMENS

The ASD No. 4 rocket nozzle assembly consists of a nozzle insert bonded into a molded phenolic housing as shown in Figure 4. All of the nozzle inserts fabricated during this report period were reinforced with plies perpendicular to the nozzle axis and were machined from blank moldings or laminates.

The method used to fabricate the nozzle insert blanks depended on the type of resin. Whenever possible, a high density, cylindrical blank was molded under high pressure in a compression mold. Some resin systems could not be cured while confined in a closed mold due to the release of excess volatiles which resulted in blistering and delamination. Materials containing such resins were laminated in an open laminating fixture which allowed the escape of excess volatiles during cure.



NOZZLE INSERT OD MACHINE FOR SLIDE FIT INTO MOLDED HOUSING
INSERT BONDED INTO HOUSING WITH HAPEX 1208 (CATALYST 1213 14 PHR), CURE 1 HOUR AT ROOM TEMP
1 HOUR AT 200°F

Figure 4. ASD No. 4 Rocket Nozzle Assembly

The nozzle housings were molded from MX2625, a heat-resistant silica fiber and mineral-filled phenolic.

Cylindrical nozzle insert blanks were molded and postcured using the conditions listed in Table VIII. All of the internal dimensions of the inserts are machined with a carbide tool. The final configuration is then machined to the ASD No. 4 dimensions. Figure 5 shows a typical machined nozzle insert. A diamond tool on a tool post grinder is used for machining.

The density of the finished insert is determined by comparing its weight with that of an insert of known density molded from general purpose phenolic.

Nozzle inserts and nozzle housings are bonded together using Hapex 1208* containing 14-percent hardener. The bond is cured 1 hour at room temperature and 1 hour at 200°F.

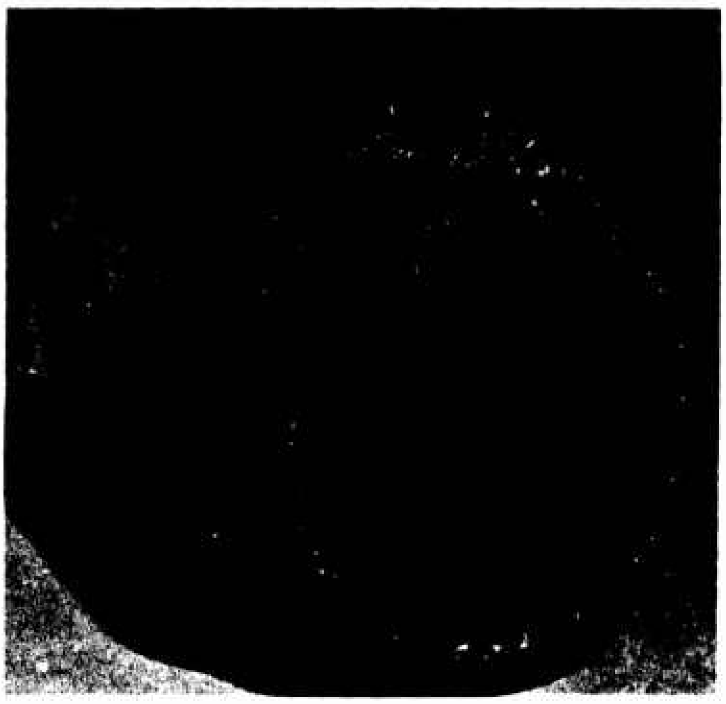


Figure 5. Completely Machined ASD No. 4 Rocket Nozzle Insert (91LD phenolic resin-Thornel 40 graphite fabric) (HAC Photo 4R01591)

*Hastings Plastics, 1704 Colorado Blvd., Santa Monica, California.

SECTION V

SPECIFIC SPECIMEN PROCEDURES

Certain specimens were fabricated by methods other than described in the previous section. Detailed procedures are listed below and are grouped according to specimen type.

1. PELLET SPECIMENS

Data Sheet No. 440 91LD-silicon carbide whiskers-carbon cloth CCA-1

The AFML Project Engineer requested the molding of a 91LD phenolic resin - silicon carbide whisker - carbon cloth CCA-1 composite. This composite was to have the same volume-percent of resin and reinforcement as one containing 35 weight-percent 91LD resin and 65 weight-percent carbon cloth CCA-1. Additionally, the volume-percentages of silicon carbide whiskers and carbon cloth CCA-1 were to be equal.

A. Density of Silicon Carbide Whiskers

The density of silicon carbide whiskers was first determined with a pycnometer and toluene as follows:

- The pycnometer was filled with toluene at 25°C. The volume of this toluene (V_1) was found by dividing the known density of toluene into the weight of liquid in the pycnometer.
- The pycnometer was emptied and dried and then an amount of silicon carbide whiskers was placed inside.
- The weight of whiskers was found and the pycnometer refilled with toluene at 25°C.
- The volume of the added toluene (V_2) was found by dividing the density of toluene into weight of added toluene.
- The volume of toluene displaced by the whiskers was found by subtracting V_2 from V_1.
- The volume of the whiskers is equal to the volume of the displaced toluene. Dividing this volume into the weight of whiskers gave a density of 3.2 gm/cc.

The amounts of silicon carbide whiskers and carbon cloth CCA-1 needed for the requested specimen were then calculated. Density values of 1.26 gm/cc and 1.84 gm/cc were used for 91LD resin and carbon cloth CCA-1, respectively.

B. Volume Composition of a 35 Weight-Percent 91LD Resin -65 Weight-Percent Carbon Cloth CCA-1 Composite

The volume-percent of resin and reinforcement have been previously calculated for composites containing 35 weight-percent 91LD resin and 65 weight-percent carbon cloth. These values are as follows:

- Volume-percent 91LD Resin = 44.1
- Volume-percent Carbon Cloth CCA-1 = 55.9

C. Calculation of Weight Composition of a 91LD-Silicon Carbide - Carbon Cloth Composite

1. For a 1 cc specimen containing 44.1 volume-percent 91LD, 27.95 volume-percent silicon carbide whiskers and 27.95 volume-percent carbon cloth, the weight of resin is found as follows:

$$W_{Resin} = D_{Resin} \times V_{Resin} = 1.26 \text{ gm/cc} \times 0.441 \text{ cc} = 0.556 \text{ gm}$$

2. Similarly, for the weights of whiskers and cloth:

a. $W_{SCW} = D_{SCW} \times V_{SCW} = 3.2 \text{ gm/cc} \times 0.2795 \text{ cc}$

$= 0.894 \text{ gm}$

b. $W_{Cloth} = D_{Cloth} \times V_{Cloth} = 1.84 \text{ gm/cc}$

$\times\ 0.2795 \text{ cc} = 0.514 \text{ gm}$

3. The total weight (and density) of the composite is

$$W_{Comp} = W_{Resin} + W_{SCW} + W_{Cloth} = 0.556 \text{ gm} + 0.894 \text{ gm} + 0.514 \text{ gm}$$

$$W_{Comp} = 1.964 \text{ gm}$$

4. The weight-percent of 91LD resin, silicon carbide whiskers and carbon cloth can then be calculated.

a. Weight-percent Resin $= \dfrac{0.556 \text{ gm}}{1.964 \text{ gm}} \times 100 = 28.3$

b. Weight-percent SCW = $\frac{0.894 \text{ gm}}{1.964 \text{ gm}}$ x 100 = 45.5

c. Weight-percent Cloth = $\frac{0.514 \text{ gm}}{1.964 \text{ gm}}$ x 100 = 26.2

D. Calculation of Weight of a 3/4-inch Diameter x 5/8-inch Pellet Specimen Having a Density of 1.96 gm/cc

$$W_{Pellet} = D_{Pellet} \times V_{Pellet} = 1.96 \text{ gm/cc} \times 4.525 \text{ cc} = 8.869 \text{ gm}$$

E. Weight of Silicon Carbide Whiskers and Carbon Cloth Required for a 3/4-inch Diameter x 5/8-inch Composite Containing 45.5 Weight-Percent Whiskers and 26.2 Weight-Percent Cloth

The weights of silicon carbide whiskers and carbon cloth CCA-1 for a required pellet weighing 8.869 gm are as follows:

- Silicon Carbide Whiskers . . . . 4.035 gm
- Carbon Cloth CCA-1 . . . . 2.324 gm

For a composite of alternating plies of silicon carbide whiskers and cloth, one-half the volume-percent of resin must be coated on the whiskers. The remaining half must be coated on the carbon cloth. The weight-percent of resin required for coating whiskers and cloth with equal volumes of resin was determined by first calculating resin content of these materials after postcure. The amount of volatiles in 91LD resin lost from drying, B-staging, molding and postcuring averages between 7-8 weight-percent. This weight-percent must be added to the final resin content to obtain the desired resin content for coating the two reinforcements.

F. Calculation of Weight-Percentage of 91LD Resin Required to Impregnate Silicon Carbide Whiskers and Carbon Cloth CCA-1

1. For a 1 cc composite, the weight of resin on silicon carbide whiskers is

$$W_{Resin(SCW)} = D_{Resin} \times V_{Resin(SCW)} = 1.26 \text{ gm/cc} \times 0.2205 \text{ cc} = 0.278 \text{ gm}$$

2. The weight of silicon carbide whiskers in the composite is

$$W_{SCW} = D_{SCW} \times V_{SCW} = 3.2 \text{ gm/cc} \times 0.2795 \text{ cc} = 0.894 \text{ gm}$$

3. To find the weight of the impregnated whiskers:

$$W_{Resin(SCW)} + W_{SCW} = 0.278 \text{ gm} + 0.894 \text{ gm} = 1.172 \text{ gm}$$

4. Therefore, the weight-percent resin on the silicon carbide whiskers is

$$\text{Weight-Percent} = \frac{W_{Resin(SCW)}}{W_{Resin(SCW)} + W_{SCW}} \times 100 = \frac{0.278 \text{ gm}}{1.172 \text{ gm}} = 23.7$$

5. Similarly, for a 1 cc composite, the weight-percent of resin on carbon cloth is 35.1.

6. The weight-percents of resin required to coat silicon carbide whiskers and carbon cloth were then calculated:

	Weight-Percent Resin After Post Cure	Weight-Percent Resin Required For Coating
Silicon Carbide Whiskers	23.7	31.7
Carbon Cloth CCA-1	35.1	43.1

A 3/4-inch diameter pellet was fabricated with 91LD resin, whiskers and carbon cloth. The cloth was coated using the spatula coating technique. The silicon carbide whiskers were coated by the soaking process described in Section III under Types of Impregnation. The pellet was then successfully molded, postcured, machined, and shipped to AFML.

- <u>Data Sheet Nos. 473-1a and 473-2a</u> — <u>Polyphenylene sulfide (sodium sulfide curing agent) — carbon cloth</u>

- <u>Data Sheet Nos. 473-1b and 473-2b</u> — <u>Polyphenylene sulfide (p-toluenesulfonic acid and xylylene glycol curing agents) — carbon cloth</u>

Refer to ROCKET NOZZLES, Data Sheet Nos. 468-1a, 468-1b and 469.

<u>Data Sheet No. 478</u> — <u>DEN 438 — polyaminoborane-Refrasil cloth</u>

Polyaminoborane cannot be used by itself as a resin matrix in making reinforced composites. The AFML Project Engineer, therefore, requested composites in which polyaminoborane was incorporated as a filler. The material is light and fluffy and has a high bulk factor. Large amounts of the powder are incorporated in the resin carrier only with great difficulty. The pellet specimens requested were to contain the following composition:

- DEN 438 resin — 25 weight-percent
- Polyaminoborane filler — 15 weight-percent
- Refrasil C100-48 cloth — 60 weight-percent

The weight of polyaminoborane required was 60 percent of the weight of DEN 438 resin.

The weights of the components are calculated as follows. A piece of cloth is weighed which is large enough to be blanked into the required number of plies. From the weight of cloth, the weight of filler is determined (weight of filler/weight of cloth as 15/60). The amount of catalyzed resin needed can then be found (weight of catalyzed resin/weight of filler as 25/15).

The polyaminoborane filler was added to catalyzed DEN 438 resin in the proper proportions and thoroughly blended using a mortar and pestle. This blending process broke down the particles of filler fine enough for the filler to be deposited in the interstices of the cloth. Failure to do this results in the powder flaking off the cloth during blanking of the plies. The resulting paste was thinned with acetone to a consistency suitable for coating the cloth. After coating, the cloth was air dried and B-staged prior to blanking into 2-inch diameter plies. A 2-inch diameter x 5/8-inch thick disc was then successfully molded.

The 2-inch diameter disc was not postcured in an oven but in the 2-inch diameter mold under 3300 psi pressure. DEN 438 resin appears to soften and weaken in the upper temperature range of the postcure. This allows any entrapped volatiles to "blow" the part as they seek to escape. The use of high pressure during postcure prevents "blow-up" during the "thermoplastic" stage of the resin.

The resin and filler content for the cured and postcured specimens were calculated as indicated below

1. Cloth of a known weight was uniformly coated with an exact amount of resin-filler blend containing a known weight of filler.

2. The resin content was found at each stage of the prepreg's preparation by first finding the weight-percent of reinforcement and filler. The sum of these components when subtracted from 100 percent gave the weight-percent of resin.

3. New weights of reinforcement and filler were calculated after the prepreg was cut or blanked into plies. The weight-percentages of each component was multiplied by the charge weight of the prepreg.

4. The resin content after molding and postcure was found using the procedures in Step No. 2.

Three 3/4-inch diameter x 1/2-inch pellet specimens were machined from the postcured disc. One of the pellets cracked severely shortly before shipment to AFML and was not included. The high filler content in the resin matrix (approximately 60 weight-percent) resulted in the specimens having poor interlaminar shear strength.

- Data Sheet No. 481 — 91 LD – poly(perfluorophenylene) – graphite cloth
- Data Sheet No. 484 — 91 LD – bisbenzimidazo-benzophenanthroline – graphite cloth

91 LD resin-graphite cloth composites were molded using poly(perfluorophenylene) and bisbenzimidazobenzophenanthroline (BBB polymer) as fillers. The amounts of cloth, filler and resin used were calculated in the same manner as described for the DEN 438-polyaminoborane-Refrasil cloth composite. The proportions used, however, were 15/55 for filler to cloth and 30/15 for resin to filler.

The filler was intimately mixed into the resin using a mortar and pestle, and acetone was added to thin the resulting paste to coating consistency. The resin-filler acetone blend was then spatula coated on the dried, graphite cloth. The impregnated cloth was air dried, oven dried, B-staged and blanked into plies prior to molding.

Pellets containing poly(perfluorophenylene) filler were individually molded because there was insufficient filler for this series of specimens to mold a 2-inch diameter disc. Pellets containing BBB polymer were machined from a 2-inch diameter disc, however, since there was sufficient filler. Additionally, six 1/4-inch diameter pellets were molded to determine the percentage weight loss of poly(perfluorophenylene) during postcure. Three of these small pellets were prepared from 91 LD resin powder and three from a 2:1 mixture, by weight, of 91 LD resin powder and poly(perfluorophenylene).

The poly(perfluorophenylene) and pure 91 LD resin pellets were postcured in the same oven using the standard B-1 schedule (72 hours from 275° to 400°F, 4 hours at 400° F). The pure resin slugs showed the usual amount of weight loss. The composites containing resin and filler either showed no weight loss or gained slightly. A possible explanation may be that the 91 LD and oxygen from the air reacted with end groups on the poly(perfluorophenylene) chain. However, without knowing either the formula or the percentage composition of the filler polymer, this explanation cannot be proven. Final resin and filler contents were not reported on the data sheet because of this phenomenon.

The 2-inch diameter disc containing BBB polymer was slightly cracked when removed from the mold. The disc was cut into three equal pie shaped pieces and each one contained a small crack parallel to the plies. Another composite could not be molded since there was sufficient filler at this time for only one. The three segments were postcured between pieces of aluminum plate held together by C-clamps. The pressure exerted by the clamps prevented further "blow-up." The postcured segments were machined into pellet specimens which appeared to be satisfactory.

The weight loss in postcure was normal for a 2-inch diameter, 30 weight-percent 91 LD resin composite. Therefore, all weight loss was attributed to the resin and none to the BBB polymer which was assumed to remain constant in weight. Resin and filler contents listed on Data Sheet No. 484 were calculated on this basis, in the same manner as described for the DEN438 -polyaminoborane-Refrasil cloth composite.

- <u>Data Sheet No. 482</u> — <u>PBI-carborane – Refrasil cloth</u>
- <u>Data Sheet No. 483</u> — <u>91LD – PBI-carborane – graphite cloth</u>
- <u>Data Sheet No. 485</u> — <u>Poly (α, α'-diphenyl-p-xylylidine) – graphite cloth</u>
- <u>Data Sheet No. 486</u> — <u>Poly (α, α'-diphenyl-m-xylylidine) – graphite cloth</u>

PBI-carborane, poly (α, α'-diphenyl-m-xylylidine) and poly (α, α'-diphenyl-p-xylylidine) resins were found to be soluble in N-methyl 2-pyrrolidone (NMP). Lacquers were prepared from each resin and NMP and coated on small pieces of cloth. These strips were vacuum dried at 300° F for several hours and then oven dried at 400° F to remove any remaining NMP. This solvent has a high boiling point (395° F) and is not easily removed from coated cloth. Traces remaining can cause a part to "blow up" when the prepreg is molded at very high temperatures (500° – 700° F).

After molding successfully 3/8-inch diameter x 1/4-inch discs with each of the resin lacquers, 3/4-inch diameter x 5/8-inch specimens were molded. Because of the small amount of poly (α, α'-diphenyl-xylylidine) resins received, their lacquers were coated directly on blanked 3/4-inch diameter plies of graphite cloth to minimize waste.

The poly(α,α'-diphenyl-xylylidine) resins appear to be thermoplastic since they continue to flow slowly under prolonged exposure to heat and pressure. Therefore, the pellet specimen molded from each type was not postcured prior to machining and shipment.

A 2-inch diameter x 5/8-inch thick PBI-carborane-Refrasil cloth disc was similarly prepared by coating the NMP-resin lacquer on blanked plies of cloth. Test pieces of this type composite were postcured in both argon and air using the temperature cycle previously used on a carborane resin-asbestos composite.* The maximum temperature reached in this cycle is 800° F. The piece exposed to air was badly oxidized and the piece postcured in argon while not oxidized was severely cracked. A second test was conducted using argon and a maximum postcure temperature of 700° F. The postcured sample did not crack but was very easily delaminated. Because of this possibility of delamination, the 2-inch diameter disc was not postcured. Three pellet specimens were machined from the unpostcured disc but in spite of precautions, one of the pellets broke before being completed. Even without postcure, the disc had poor interlaminar shear strength. The two remaining pellets were shipped to AFML.

Composites were prepared containing 91 LD resin (30 weight-percent) PBI-carborane resin as filler (15 weight-percent) and graphite cloth G1550 (55 weight-percent). The small amount of PBI-carborane resin remaining after previous attempts to mold this material, did not allow preparing a 2-inch diameter disc. 91-LD resin varnish and PBI-carborane resin filler were intimately mixed together in the required proportions using a mortar and pestle. This mixture was coated on previously blanked 3/4-inch diameter plies of graphite cloth to minimize waste of filler. Three 3/4-inch diameter x 1/2-inch pellet specimens then were successfully molded and postcured.

- <u>Data Sheet No. 520</u> <u>Abchar 413[1] – Abchar 700[2] – carbon cloth</u>
- <u>Data Sheet No. 522</u> <u>Abchar 412B[3] – Abchar 700[2] – carbon cloth</u>

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*The temperature was raised to 250° F over 16 hours, then held at 250° F for 12 hours. The temperature was then raised at the rate of 50° per 12 hours to 800° F. After being held at 800° F for 24 hours, the part was cooled to below 200° F before removing.

[1]Information on this resin is given on page 38

[2]Information on this resin is given on page 36

[3]Information on this resin is given on page 37

A 3-1/2-inch diameter x 5/8-inch disc was molded and postcured using Abchar 413[1] resin, Abchar 700[2] filler and carbon cloth. A second 3-1/2-inch diameter x 5/8-inch disc was molded and postcured using Abchar 412B,[3] Abchar 700[2] filler and carbon cloth. Fabrication procedures for both were the same regardless of the resin system. The carbon cloth was oven dried to remove residual moisture and then weighed. The weight of Abchar 700[2] powder required was calculated using the ratios 15/55 for filler to cloth and 30/15 for resin solids to filler.

A Waring Blendor was used to intimately mix Abchar 700[2] into the resin solution. The materials were blended for 15 minutes after which acetone was added to thin the mix to coating consistency. The carbon cloth was spatula coated, air dried, oven dried, B-staged, molded and postcured using the conditions indicated in Table VII, Fabrication Details—Pellet Specimens.

Resin and filler contents for the cured and postcured specimens were calculated in the same manner as described for the DEN 438-poly-aminoborane-Refrasil cloth composite. The volume-percent voids in the resin matrix was calculated from the following formulas.

1. $$D_M = \frac{\Sigma W}{\Sigma V} = \frac{W_M}{V_{VFR} + V_C + V_F}$$

since $V = \frac{W}{D}$,

$$D_M = \frac{W_M}{\frac{W_R}{D_{VFR}} + \frac{W_C}{D_C} + \frac{W_F}{D_F}}$$

where

D_M = density of molding

W_M = total weight of components in the molding

V_M = total volume of components in the molding

V_{VFR} = volume of void filled resin

V_C = volume of cloth

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[1] Information on this resin is given on page 38

[2] Information on this resin is given on page 36

[3] Information on this resin (Batch C2943-72A) is given on page 37

V_F = volume of filler

D_{VFR} = density of void filled resin

D_C = density of cloth

D_F = density of filler

W_R = weight of resin

W_C = weight of cloth

W_F = weight of filler

2. Volume percent voids in resin $= \frac{D_R - D_{VFR}}{D_R} \times 100$

where

D_R = density of void-free resin

D_{VFR} = density of void filled resin

Calculating the final resin, reinforcement and filler content as well as the volume-percent voids in the resin matrix was contingent on one factor. This factor was that Abchar 700[1] did not lose any weight during postcure. Previous work reported in Technical Report AFML-TR-66-75, Part II, Page 30 indicated this assumption to be correct.

Six pellet specimens were machined from each 3-1/2 inch diameter disc and shipped to AFML.

- <u>Data Sheet No. 524</u> <u>Teflon 30-boron nitride fibers</u>
- <u>Data Sheet No. 526</u> <u>Teflon 30</u>

Teflon 30 and Teflon 30-boron nitride fiber composites were molded in the following manner. The required charge weights were calculated from the material in the preform mold. A specimen preform thickness of 5/8 inch was used in calculating the charge weight of a 1/2-inch thick pellet. Similarly, a preform thickness of 2-1/2 inches was used for a 2-inch thick cylinder.

The entire charge weight for a specimen was preformed in one operation in a cold mold at 5000 psi. The mold was then heated to 700° F as rapidly as possible and the molding material sintered.

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[1]Information on this resin is given on page 36

The high bulk factor of the material necessitated a special mold for the 2-inch thick cylinders. This mold was 13 inches and could be separated into two pieces, a bottom section 4 inches high and a top section 9 inches high. After preforming, the top section was removed and the part was sintered in the bottom section. A shorter punch was used to apply pressure.

All the required specimens were prepared except for the 2-inch thick Teflon 30-boron nitride composites. A problem occurred when excessive escaping volatiles extruded molding material out the bottom of the mold and raised the mold up from the press platen. This phenomenon prevented the obtaining of satisfactory parts.

The Teflon 30 and Teflon 30-boron nitride fiber composites which were molded, were machined to the required dimensions. The close tolerance required for the diameter of the 2-inch thick cylinders were obtained in different ways. For the cylinder with the center hole, the hole was first drilled and the specimen mounted on a proper size mandrel passing through the hole. The cylinder was then machined to the required outer diameter. The cylinder without the hole was centerless ground to dimension. The machined parts were then shipped to AFML.

- Data Sheet Nos. 530a, b, and c — Abchar L913[1] - carbon cloth
- Data Sheet No. 531 — Abchar L1112[2] - carbon cloth
- Data Sheet Nos. 532a, b, and c — Abchar H913[3] - carbon cloth
- Data Sheet Nos. 533a, b, and c — Abchar H1013[4] - carbon cloth

The AFML Project Engineer requested the fabrication of pellet specimens from carbon cloth and polyphenylene resins with new curing agents. These resins are as follows:

1. Abchar L913 - polyphenylene (MW 1000) with 1, 3, 5-benzenetrisulfonyl chloride as curing agent
2. Abchar L1023 - polyphenylene (MW 1000) with 4, 4'-biphenyldisulfonic acid as curing agent
3. Abchar H913 - polyphenylene (MW 2000) with 1, 3, 5-benzenetrisulfonyl chloride as curing agent
4. Abchar H1013 - polyphenylene (MW 2000) with 4, 4'-biphenyldisulfonic acid as curing agent

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[1] Information on this resin is given on page 39

[2] Information on this resin is given on page 40

[3] Information on this resin is given on page 39

[4] Information on this resin is given on page 39

Abchar L913[1] was furnished as a lacquer, Abchar H913A[1] and Abchar H1023[1] as dry powders. Abchar L1023 was also in powder form even though the resin is low molecular weight. A lacquer could not be prepared because the polymer and curing agent are not soluble in the same solvents.

Specimens molded from Abchar L913[1] were prepared using standard impregnating and molding procedures and used untreated carbon cloth. The dry powders were mixed with chloroform to form a slurry. This slurry was spatula coated on carbon cloth pretreated by Dr. Leroy Miller of the Materials Technology Department's Polymer and Chemical Technology Group.

The purpose of the pretreatment was to try to chemically link an organic compound to the surface of the carbon cloth. This compound, organically similar to the curing agent in the polyphenylene, would then link with the impregnating resin during molding. Use of pretreatment should greatly improve the adhesion of the resin to the cloth if the chemical linkage takes place.

Dr. Miller stated that the pretreatment would lose weight during cure and postcure. This loss of weight is subtracted from the resin weight rather than the reinforcement weight. The resin content as reported is the difference between 100 weight-percent and the reinforcement weight-percent. This method is used since the reinforcement weight remains constant during cure and postcure. The weight loss of the composites cannot be correctly apportioned between the resin and pretreatment.

After spatula coating, the prepreg was air-dried for 30 minutes and oven-dried at 160° F for 10 minutes. The prepreg was then blanked into 1-inch diameter plies and molded at 500° F and 3300 psi pressure for 2 hours.

The composites were postcured at considerably higher temperatures than usually used for polyphenylenes. The standard polyphenylene postcure cycle consists of a gradual 108 hour temperature rise from 275° to 550°F. This cycle was modified at the suggestion of Dr. Bilow and Dr. Miller to a temperature rise from 275° to 750°F over 120 hours. They believe that optimum resin properties will not be obtained with these curing agents at lower postcure temperatures. An argon atmosphere is used in both cycles to prevent excessive resin oxidation at elevated temperatures.

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[1]Information on these resins is given on page 39

Pellets were machined from the postcured composites. The interlaminar shear strength was poor for several of the moldings, however, and plies readily broke off during machining. The thickness of the remaining parts was well below tolerance and the specimens had to be discarded. Those specimens which were successfully machined were shipped to AFML.

Abchar L1023 polyphenylene resin could not be successfully molded. A replacement low molecular weight resin catalyzed with 4, 4'-biphenylsulfonyl chloride was furnished as a lacquer and designated as Abchar L1112.[1] Six carbon cloth discs, 1-inch diameter x 5/8-inch were then molded with this lacquer using a spatula coated prepreg. However, all specimens severely cracked during postcure and only one pellet could be machined from them. Cracking was probably caused by the large amount of volatiles lost by the discs during postcure. This weight-loss ranged from 16-20 percent. The 120 hours of the postcure, from 275° to 750° F was insufficient time to allow such a volume of gas to escape without fracturing the parts.

Data Sheet No. 537	Polyimidazopyrrolone (Pyrrone) - carbon cloth

Refer to ROCKET NOZZLES, Data Sheet No. 536

- Data Sheet No. 539 a&b — SC1008 - Thornel 25 graphite fiber tape
- Data Sheet No. 541/552 — SC1008 - graphite yarn WYB 85 1/2 tape
- Data Sheet No. 543/553 and 543b — SC1008 - carbon yarn VYB 70 1/2 tape
- Data Sheet No. 545 — Skybond 703 - Thornel 25 graphite fiber tape
- Data Sheet No. 547 — DP-25-10 - Thornel 25 graphite fiber tape
- Data Sheet No. 548 — SC1008 - quartz yarn
- Data Sheet No. 549 — p-Phenylphenol phenol formaldehyde - Thornel 25 graphite fiber tape

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[1] Information on this resin is given on page 40

- <u>Data Sheet No. 550</u> — <u>Abchar 413[1] - Thornel 25 graphite fiber tape</u>
- <u>Data Sheet No. 551</u> — <u>DP-4-31 - Thornel 25 graphite fiber tape</u>

Refer to ROCKET NOZZLES, Data Sheet Nos. 538, 540/552, 542/553, 542b and 546

<u>Data Sheet No. 555</u> — <u>Polyphenylene sulfide - carbon cloth</u>

A 2-inch diameter x 5/8-inch disc was molded containing Phillips Petroleum polyphenylene sulfide resin and carbon cloth. The specimen was prepared by molding a dry powder layup at 650° F (the manufacturer's recommended cure temperature) for 2 hours. The resulting disc was then cut into three equal pie shaped pieces to minimize the possibility of "blow-up" during postcure. These pieces were postcured using the standard Hughes polyphenylene temperature cycle (Table VII, Fabrication Details—Pellet Specimens).

In spite of the precautions taken, the three pieces had increased 28.6 percent in thickness when removed from the oven. Escaping volatiles during postcure probably acted like blowing agent in foam since the resin appears to be thermoplastic at high temperature.

Based on the assumption the resin could flow again and compress if reheated under pressure, pieces of composite were put back in the mold. The mold was heated to 600°F while the parts were under 3300 psi pressure. After 1 hour at temperature, the parts were cooled still under pressure to below 200°F. When removed from the mold the pieces had bonded together and the resulting part was approximately the thickness of the original molding. Three pellet specimens were machined from the "remolded disc."

- <u>Data Sheet No. 559</u> — <u>91 LD - bisbenzimidazobenzophenanthroline - carbon cloth</u>

Refer to ROCKET NOZZLES, Data Sheet Nos. 562 and 565

<u>Data Sheet No. 560</u> — <u>91 LD - Thornel 40 graphite fabric</u>

Refer to ROCKET NOZZLES, Data Sheet No. 566

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[1]Information on this resin is given on page 38

Data Sheet No. 561   Abchar 412B[1] - carbon cloth

Refer to ROCKET NOZZLES, Data Sheet No. 567

2. ROCKET NOZZLES

Data Sheet No. 408   F171 - carbon cloth

One laminated square, 2 inches x 2 inches x 1/2 inch, and one ASD No. 4 rocket nozzle specimen were prepared using F171 polyarylene-phenolic resin and carbon cloth. Both specimens had resin contents below the requested 40 weight-percent value (laminated square - 27.7 weight-percent; nozzle - 35.4 weight-percent). These items could not be remade because more resin varnish cannot be obtained. The vendor manufacturing this resin system reports that their supplier is no longer synthesizing the basic polymer.

Sufficient F171 resin varnish was on hand to prepare one laminated square and one nozzle. The reinforcements for the laminated square and nozzle were coated with all the resin varnish on hand using standard procedures. However, these prepregs when B-staged, contained several percent less resin than desirable prior to molding.

The molded nozzle blank broke in two while being removed from the mold. After postcure, the pieces were bonded together with Hapex 1208 adhesive using the nozzle mold as a holding fixture. Machining of the nozzle was successfully accomplished by the short nozzle housing - polyglycol technique. This technique is as follows:

- A rocket nozzle housing was modified to allow its use as a machining fixture. To accomplish this, the flange of the housing was removed and the housing's length trimmed to 2.06 inches.
- After completion of machining the step in the nozzle insert, the part was bonded into the short housing with polyglycol.
- The remaining dimensions of the insert were then machined with the possibility of further delamination minimized.
- The insert was removed from the machining fixture by heating both for one hour at 275° F and then pushing the insert out.
- Traces of polyglycol were removed from the part by careful washing in hot water.
- After drying for 2 hours at 225° F, the insert was bonded into a regular nozzle housing using standard procedures.

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[1]Information on this resin is given on page 40

- Data Sheet No. 468-1a — Polyphenylene sulfide (sodium sulfide curing agent) - carbon cloth
- Data Sheet No. 468-1b — Polyphenylene sulfide (p-toluene - sulfonic acid and xylylene glycol curing agents) - carbon cloth
- Data Sheet No. 469 — Polyphenylene sulfide (p-toluene - sulfonic acid and xylylene glycol curing agents) - Refrasil cloth

Three batches of Dow Chemical Company experimental polyphenylene sulfide resins were submitted for evaluation by AFML. These batches are as follows:

- QX-4375.4 (Lot 822-6A) without curing agent
- QX-4375.4 (Lot 822-6B) with calcium sulfide curing agent
- QX-4375.1 (Lot 822-6C) without curing agent

Dow Chemical Company recommended that Lot 822-6A resin could be cured using either sodium sulfide or calcium sulfide as curing agent. Additionally, Dow stated that Lot 822-6B which already contained curing agent and Lot 822-6C which did not, could be cured without further modification. Before starting the evaluation of these resins, Dr. Norman Bilow was consulted about other possible curing agents for these materials. He believed that some of those curing agents presently used for Abchar polyphenylene resins would also crosslink the Dow resins. With the concurrence of the AMFL Project Engineer, small samples of Lot 822-6A and Lot 822-6C were submitted to Dr. Bilow for addition of crosslinking agents. Lot 822-6B was not submitted because this resin already contained a crosslinking agent.

Dr. Bilow modified each lot of material with the following curing agents:

- 1 3, 5-benzenetrisulfonyl chloride
- p-toluenesulfonic acid monohydrate and p-xylylene glycol

Additionally, a test batch of Lot 822-6A was prepared containing one mole of sodium sulfide per mole of total halide in the resin.* All batches of resin were then evaluated by molding the resin powder without reinforcement at various temperatures and pressures. When a hard, tough resin slug was obtained, a carbon cloth composite was then attempted using a dry powder layup. A summary of results obtained with these materials is listed on the following page.

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*The percent halides present in this resin batch were listed in letter to AFML, 2 December 1965 from Dow Chemical Company

Based on these results, the AFML Project Engineer requested pellets and nozzles using the "satisfactory" combinations. Dr. Bilow prepared sufficient amount of each type of modified resin for the required specimens. Several small carbon cloth composites were then molded with Lot 822-6A resin using both curing agent systems. One of each type of composite was postcured using the standard Hughes polyphenylene resin postcure (108 hours from 275° to 550°F, 6 hours at 550°F). When removed from postcure, both types were found to have delaminated.

Two small carbon cloth composites containing Lot 822-6A resin with sodium sulfide curing agent were postcured under pressure in a mold. The composites were heated to 600° F under 5000 psi pressure, one specimen for 4 hours, the other for 24 hours. The weight loss was small for both postcures.

A 16-hour combined cure-postcure was decided upon for molding Lot 822-6A resin mixes to prevent tying up presses and molds. A mold containing a dry powder layup was placed in a 600° F press at 5000 psi late in the afternoon and then removed the following morning. Previously molded specimens containing Lot 822-6A resin were postcured similarly regardless of the curing agent used.

Summary of Results Molding Test Batches of Polyphenylene Sulfide Resins with Various Curing Agents

Dow Chemical Resin Batch Number	Curing Agent	Result of Molding Pure Resin Slug	Result of Molding Carbon Cloth CCA-1 Composite
QX4375.4 (822-6A)	Sodium sulfide (1 mole per mole total halide)	Satisfactory	Satisfactory
	1,3,5-benzene-trisulfonyl chloride	Unsatisfactory	-
	p-toluenesulfonic acid monohydrate and p-xylylene glycol	Satisfactory	Unsatisfactory
QX4375.4 (822-6B)	Used as received	Satisfactory	Unsatisfactory
QX4375.4 (822-6C)	Used as received	Satisfactory	Unsatisfactory
	1,3,5-benzene-trisulfonyl chloride	Satisfactory	Satisfactory
	p-toluenesulfonic acid monohydrate and p-xylylene glycol	Satisfactory	Marginal

Composites were labeled "unsatisfactory" when the resin did not wet or adhere to the cloth. The composite labeled "marginal" held together but the quality of the molding was poor.

Two discs, 2-inch diameter x 5/8 inch, prepared using this cure-postcure method cracked while being machined. One disc was cured using sodium sulfide while the other was cured using p-toluenesulfonic acid monohydrate and p-xylyene glycol. Care had been exercised during machining but the pellet specimens still contained large cracks. These specimens were not shipped but additional composites were molded and new acceptable pellets machined.

The reason for this cracking is unknown. However, cracking may indicate the following about composites molded using these resin systems.

- The polymer is not completely crosslinked and has poor interlaminar shear strength
- The polymer wets the reinforcement poorly with little bonding between the resin and plies of cloth
- Large stresses may be present in the discs because of the molding method. Pellet specimens machined from these discs may stress relieve themselves causing cracking

Pellets and nozzles containing Lot 822-6A resin with the requested system of curing agents have been sent to AFML for evaluation. The densities of pure resin slugs with these systems have been obtained for calculating the volume-percent voids in the composites. These densities are as follows:

- Batch QX 4375.4 (sodium sulfide curing agent) 1.32 gm/cc
- Batch QX 4375.4 (p-toluenesulfonic acid monohydrate and p-xylylene glycol curing agents) 1.36 gm/cc

Data Sheet No. 536 Polyimidazopyrrolone (Pyrrone) - carbon cloth

The AFML Project Engineer requested by phone the fabrication of two nozzle inserts and six pellet specimens from a Pyrrone varnish prepared from pyromellitic dianhydride (PMDA) and 4,4'-diaminobenzidine (DAB), both as solutions in dimethylacetamide. The specimens were to contain 35 weight-percent of resin solids and 65 weight-percent of carbon cloth CCA-1. PMDA-DAB Pyrrone resin loses very large amounts of volatiles during cure. Therefore, nozzle insert blanks could not be molded in a closed mold but only in the 1-3/4-inch x 1-3/4-inch laminating fixture. Similarly, a 3-1/2-inch diameter disc could not be molded for machining into pellet specimens. Instead, a 4-inch x 2-inch x 5/8 inch laminate was prepared.

PMDA-DAB Pyrrone varnish received from Narmco was used to fabricate the nozzle blanks and laminate. The varnish contained 10 percent resin solids and was used as received in dip coating dried carbon cloth. The cloth required eight dips to obtain the desired resin content (approximately 62 weight-percent). The cloth was air dried for 10 minutes between dips and given a 3-minute flash dry at 200° F after every other dip. Prior to molding, the resulting prepreg was dried in a 200° F oven for 12 minutes. During molding, after a 3-minute contact time, the prepreg was cured at 300° F and 1000 psi pressure for 60 minutes. The nozzle blanks and laminate were postcured from 275° to 600° F over 165 hours and at 600° F for 27 hours.

A large weight loss occurred in both nozzle blanks and laminate during cure and postcure. This resulted in the specimens containing substantially less resin than requested (20.4 weight-percent and 29.8 weight-percent for the nozzles and 13.0 weight-percent for the pellets). Additionally, the volume-percentage of voids in the resin was extremely high (74.6 percent and 70.0 percent for the nozzles and 82.3 percent for the pellets). Such a great weight loss of volatiles resulted in highly porous composites similar to high density foams.

The rocket nozzle blanks were so weak that final machining could not be completed until they were bonded into nozzle housings. No problem occurred, however, in machining pellet specimens from the laminate.

- Data Sheet No. 538 — SC1008 - Thornel 25 graphite fiber tape
- Data Sheet No. 540/552 — SC1008 - graphite yarn WYB-85 1/2 tape
- Data Sheet Nos. 542/553 and 542b — SC1008 - carbon yarn VYB-70 1/2 tape
- Data Sheet No. 546 — DP-25-10 - Thornel 25 graphite fiber tape

Work on Initial Shipment of Preimpregnated Tapes

Several hundred inches of impregnated tapes containing high modulus collimated fibers were received from AFML for the fabrication of pellets and nozzles. These tapes came in two widths, 1 inch wide for pellets and 1-3/4 inches wide for nozzles. A list of resin contents for the various pieces was furnished by AFML and ranged from 42 percent to over 70 percent. These values were checked by running Soxhlet extractions with acetone. Spot testing of various tape runs showed good agreement with the AFML results.

The first specimen molded was 1 inch x 1 inch x 3/16 inch and was made using SC1008 phenolic resin-Thornel 25 fiber tape. The tape used contained 73 weight-percent of resin and no attempt was made to obtain a specimen with the desired resin content. The molding was of excellent quality but had a resin content of approximately 50 weight-percent.

This specimen was prepared in the following manner. Pieces of 1 inch wide impregnated tape were cut 4-1/8 inches long and laid side by side to form a rectangle 4-1/8 x 4 inches. The ends of the fibers were held down on a block using masking tape. A second layer 4-1/8 x 4 inches was laid down at right angles over the first and tacked down to the first using a hot tacking iron. A piece of Mylar film was placed on the fibers during the tacking operation to prevent the resin from sticking to the iron. The layup was then cut into sixteen 1 x 1 inch squares with a blanking tool. Sufficient pieces were then placed into the 1 inch square mold with fibers in every ply oriented 90° to fibers in adjacent plies.

The number of plies needed to mold a 1-inch x 1-inch x 5/8-inch block containing 35 weight-percent resin was found in the following manner. First, the weight per unit area was obtained for unimpregnated Thornel 25 fibers. This was calculated by averaging the weights of several pieces of 1-inch x 1-inch tape after the resin had been removed by extraction. Second, the theoretical weight was calculated for a 35 percent resin content, 1-inch x 1-inch x 5/8-inch block. Finally, the number of tape plies in this molding was found by dividing 65 percent of the block weight by the weight per ply.

A second molding was made with the number of piles calculated as described above. To ensure the correct resin content, a dial micrometer was used to determine when the tape had been compressed to 0.625 inch in the mold. This squeezing of the tape to size coupled with its initial high resin content caused a large volume of resin to flow from the mold. This flow carried much of the Thornel 25 fibers with it. The fibers remaining in the molding were no longer oriented in layers but in jack-straw fashion.

This excessive resin content posed a dilemma. Decreased pressure on the part would not allow the punch to come down to the required 0.625-inch thickness before the resin gelled. Further resin advancement prior to molding would simply make matters worse. The advanced resin would be more viscous during flow and require a greater pressure to remove the excess. This pressure would then carry out more fibers.

The solution to the problem was to use tapes containing about 42-44 weight-percent resin. With proper B-staging, the flow would be held to a minimum with little or no movement of the fibers in the plies.

Two experimental moldings were prepared using 1-inch wide tape containing 41.5 weight-percent phenolic resin. The first 0.625-inch thick molding contained 30 weight-percent resin. A longer B-stage time resulted in the second part containing 33 weight-percent resin. Both parts were unacceptable since the resin content was below the allowable limit before postcure. However both parts were of excellent quality and a still longer B-stage time would have resulted in moldings with the required resin content.

Work on Second Shipment of Preimpregnated Tapes

Based on the above work, AFML sent new shipments of impregnated high modulus collimated tapes with the desired amount of resin. The first molding was made by first oven drying the plies for 60 minutes and then B-staging them for 60 minutes. This molding contained the required resin content and bidirectional fiber orientation. Flow was kept to a minimum and no loss of fibers in the flash occurred.

Work then continued in molding pellets and rocket nozzles with the new shipments of tapes. Pellets were prepared from bidirectional layups using 1-inch wide tape. Rocket nozzles were similarly prepared from bidirectional layups using 1-3/4-inch wide tape. Eight double plies, 1-3/4 x 1-3/4 inches, were blanked from a 7-1/8 x 3-1/2 inch layup for molding in the 1-3/4 inch square mold.

Two rocket nozzle blocks, 1-3/4 inch x 1-3/4 inch x 3 inches were molded oversize and both pellets and rocket nozzles machined from them. One block contained SC 1008 resin on VYB70 1/2 carbon fibers and the other, SC1008 resin on WYB 85 1/2 graphite fibers.

The tapes containing p-phenylphenol phenol formaldehyde resins averaged only 38 weight-percent resin content as received. Anything greater than a slight flow during molding would result in these specimens being out of tolerance. The tapes, therefore, were given sufficient B-stage time to almost eliminate any flow during cure. Additionally, the low resin content was insufficient to hold the tape together during blanking. After blanking, pieces of B-staged tape less than the width of a ply were removed from the blanking die. These split pieces had to be correctly oriented by carefully laying them in the mold without disturbing the arrangement of the lower plies.

Two nozzle blanks containing SC1008 resin and Thornel 25 fiber tape broke into pieces while being machined into nozzle inserts. These blanks broke although special preventative precautions had been taken during machining. The interlaminar shear strength of the composites was too poor to hold the moldings together during the machining operations.

The AFML Project Engineer was informed, and ways were discussed for salvaging these nozzle blanks. Salvage procedures were decided upon and carried out as follows:

- The pieces of the blanks were bonded together using Hapex 1208 adhesive, the same system used tc bond nozzle inserts into housings.
- The nozzle blanks were then machined using the short nozzle housing-polyglycol technique described under Data Sheet No. 408.

All requested nozzles and pellets were fabricated except for one nozzle and one pellet. There was insufficient acceptable material remaining to mold these two items.

- <u>Data Sheet No. 562</u> <u>91 LD-poly(perfluorophenylene) - carbon cloth</u>
- <u>Data Sheet No. 565</u> <u>91 LD - bisbenzimidazo - benzophenanthroline - carbon cloth</u>

91 LD resin-carbon cloth CCA-1 composites were prepared incorporating either poly(perfluorophenylene) or bisbenzimidazo - benzophenanthroline filler. Filler and resin varnish were blended together in the required ratio using a Waring Blendor. Mixtures were thinned with acetone to coating consistency and then coated on reinforcement. After air drying, oven drying and B-staging, the preimpregnated fabric was blanked into the proper size plies and molded. Specimens were then postcured, machined and shipped to AFML. No increase in weight of the 91 LD-poly(perfluorophenylene) composites took place after postcure such as had been previously noted (Data Sheet No. 481).

<u>Data Sheet No. 566</u> <u>91 LD - Thornel 40 graphite fiber</u>

A small 91 LD - Thornel 40 graphite cloth composite was molded to determine the effective thickness per ply of the reinforcement. The thickness per ply was found to be 0.005 inch. Using this value, the minimum amount of cloth needed to mold two nozzles was slightly greater than the amount received from AFML. One nozzle and two 2-inch diameter discs were then molded to allow for the testing of both rocket nozzle and pellet specimens.

<u>Data Sheet No. 567</u> <u>Abchar 412B[1] — carbon cloth</u>

Hughes Aircraft Company was supplied with 2 pounds of polyphenylene resin containing xylylene glycol curing agent (Abchar 412B). This resin was manufactured by Allied Chemical and supplied to Hughes under Contract AF 33(615)-5361. Test specimens were prepared with this resin and were satisfactory.

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[1]Information on this resin is given on page 40

Two nozzle blanks and a 3-1/2 inch diameter x 5/8-inch disc were postcured and machined. One nozzle blank broke during the machining operation although the specimen contained a 15/32-inch bolt through the pilot hole. This specimen apparently had poor interlaminar shear strength. Also, the pellet specimens machined from the 3-1/2-inch diameter disc appeared to contain hairline fractures. However, these specimens had machined satisfactorily and showed no tendency to break during this operation.

3. MISCELLANEOUS TYPES OF SPECIMENS

Data Sheet No. 462 | F171 - carbon cloth | Laminated square

Refer to ROCKET NOZZLES, Data Sheet No. 408

Data Sheet No. 525 | Teflon 30 | Cylinder

Refer to PELLET SPECIMENS, Data Sheet No. 524 and 526

Data Sheet No. 572a&b | Thermogravimetric Analysis Specimens

Six thermogravimetric analysis specimens each weighing approximately three grams were prepared using the following resins:

- Phillips Petroleum polyphenylene sulfide
- Abchar L913[1]
- Abchar 412B (xylylene glycol curing agent)[2]
- 91 LD
- 91 LD with poly(perfluorophenylene)
- 91 LD with BBB polymer

The Abchar L913, Abchar 412B and 91 LD resin lacquers were vacuum dried to a powder prior to molding. 91 LD resin powder was blended in the proper proportions with the required fillers and then intimately mixed. After molding, all resin slugs were postcured using the appropriate cycles.

[1] Information on this resin is given on page 39

[2] Information on this resin is given on page 40

APPENDIX

FORMULATIONS USED WITH MULTIPART RESIN SYSTEMS

Material	Parts by Weight
DEN 438	
DEN 438 (Epoxy novolac)	100
Methyl nadic anhydride	100
Benzyldimethylamine	1

RESIN SYNTHESES

All resins were synthesized by the Polymer and Chemical Technology Group of the Materials Technology Department under the direction of Dr. Norman Bilow.

Abchar 700

Intractable Polyphenylene

Batch No. C2426-15C

Intractable polyphenylene isolated from a cationic oxidative polymerization of an equimolar mixture of biphenyl and terphenyl was repeatedly washed with concentrated aqueous hydrochloric acid until the wash was virtually colorless. The polymer was then extracted continuously with boiling trichlorobenzene to remove any residual soluble polyphenylene. It was then washed with naphtha and dried with heat and vacuum. This resin is equivalent to Batch C1414-27 of Abchar 700 previously prepared.

Abchar 412

Polyphenylene

Batch No. C2813-10A

The curing agent was prepared by mixing p-xylylene glycol (recrystallized) (50 gm), p-toluenesulfonic acid monohydrate (15 gm) and chlorcform (300 ml) and then slurrying these ingredients cold for 1/2 hour. The mixture was heated to reflux and reacted for 20 hours with a water trap. Seven milliliters of water were collected.

The lacquer was prepared by first adding 50 gm of polyphenylene (M. P. = 140-182°C) and 50 gm of polyphenylene (M. P. = 250-285°C) to 200 ml of trichloroethylene, while stirring. The average molecular weight of this resin mix was approximately 1400. The mixture was slurred cold for 2 hours and then heated to reflux to react for 1-1/2 hours. Curing agent was added and the new mixture refluxed for 20 hours at 70°C. After filtering, the yield was 1368 gm (16 percent solids).

Abchar 412B[1]

Polyphenylene

Batch No. C2813-19

p-Xylylene glycol (50 gm), p-toluenesulfonic acid monohydrate (16 gm) and chloroform were heated at reflux for 20 hours while removing water continuously. Most of the chloroform was then removed under vacuum, and 20 ml of p-dioxane was added. Residual chloroform was then removed at 50°C under vacuum.

A second solution was prepared by dissolving 60 gm of polyphenylene (M. P. 140°-182°C., M. W. ≈ 900, C/H = 1.57) and 40 gm of fully aromatic coal tar pitch (Koppers Chemical Co. 40111) in p-dioxane (200 ml). After heating at reflux for 2 hours, the solution was cooled to 70°C, and the xylylene glycol solution was added. The mixed lacquer was heated at 70°C for 3-1/2 hours. It weighed 826 gm and contained 18 percent solids.

Abchar 412B

Polyphenylene

Batch No. C2943-72A

Xylylene glycol curing agent was prepared from 90 gm of the glycol, 27 gm of p-toluenesulfonic acid monohydrate by refluxing the two compounds in chloroform for 20 hours while removing water azeotropically.

The above solution was then added to a solution of polyphenylene (170 gm, ≈ 1000 M. W.) in trichloroethylene (360 ml), and the mixture was reacted for 20 hours at 70°C. After filtration the lacquer weighed 1543 gm and contained 17.5 percent solids.

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[1] Modified for fi.ament winding application

Abchar 413

Polyphenylene

Batch No. C2943-72B

A xylylene glycol curing agent was prepared from 65 gm of the glycol and 20 gm of p-toluenesulfonic acid monohydrate in chloroform (400 ml) by refluxing 20 hours while removing water azeotropically.

The above solution was added to a solution of polyphenylene (200 gm, 1200 M. W.) in trichloroethylene (400 ml), and the mixture was heated at 70°C for 20 hours. The filtered lacquer weighed 1334 gm and contained 20 percent solids.

Abchar 412

Polyphenylene

Batch No. C2943-74A

The curing agent was prepared by mixing p-xylylene glycol (recrystallized) (125 gm), p-toluenesulfonic acid monohydrate (38 gm) and chloroform (900 ml) and then refluxing these ingredients for 20 hours. Using a water trap, 16 ml of water were collected.

The lacquer was prepared by adding 250 gm of polyphenylene (M. W. ≅ 1200; M. P. = 150°-250°C) to 500 ml of trichloroethylene and slurring the mixture cold for 1/2 hour. The mixture was then heated to reflux and 2 hours later the curing agent added. The lacquer was reacted for 20 hours at 70°C then filtered through a centrifugal filter at 60°C. The yield was 1897 gm (19. 8 percent solids) with very little residue.

Abchar H913A (formerly Abchar H913 resin)

Polyphenylene

Batch No. C2943-81B

A mixture of polyphenylene (72 gm, M.W. $\geq$1500) and 1,3,5-benzenetrisulfonyl chloride (27 gm) in a ball mill was dried at 125°C for 1 hour. The mixture was then milled for 2 days with stainless steel balls. Milling was stopped twice during this time to break up the cake which formed on the walls of the mill. Weight of recovered solids was 95 gm.

Abchar L913

Polyphenylene

Batch No. C2943-82A

A solution of polyphenylene (60 gm, M.W. = 900) and 1,3,5-benzenetrisulfonyl chloride (20 gm) in chloroform (240 ml) was heated under reflux for 19 hours. The solution was filtered, using a small quantity of additional chloroform to wash the filter. Weight of lacquer was 544 gm.

Abchar H1023 (formerly Abchar H1013 resin)

Polyphenylene

Batch No. D1081-19-2

Polyphenylene (60 gm, M.W. $\geq$1500) and dry, recrystallized 4,4'-biphenyldisulfonic acid (40 gm) were ground in a ball mill for 2 days. The mixture was passed through a 100-mesh screen and dried in a vacuum oven for 2-1/2 hours at 115-130°C. It weighed 99.2 gm. Since the powder softened only partially at 200°C and hardened above 240°C, it was not advanced further before use.

Abchar L1112

Polyphenylene

Batch No. D1081-22-1

A solution of polyphenylene (104.0 gm, M.W. ≅900) and 4,4'-biphenyldisulfonyl chloride (52.0 gm) in 1,1,2,2-tetrachloroethane (624 ml) was heated under reflux for 8 hours, and a small quantity of undissolved solid material (estimated at 2 to 4 gm) was filtered off. Weight of lacquer was 1145 gm.

Abchar 412B

Polyphenylene with xylylene glycol

Batch No. 31382-37

This resin was manufactured by Allied Chemical Corporation. The method of manufacture is described in the final report for Air Force Contract No. AF 33(615)-5361, "Polyphenylene Resin (Aromatic)," AFML-TR-68-55.

TABLE :

PROPERTIES OF PELLE?' SPECIMENS

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent: Resin	Composition, Weight-Percent: Reinforcement	Composition, Weight-Percent: Volume-Percent Voids in Resin	Description of Material
440	9-28-SCWC	1.62	63	30.5 weight-percent (46.7 volume-percent)	25.4 weight-percent (26.6 volume-percent) (carbon cloth) 44.1 weight-percent (26.6 volume-percent) (whiskers)	28.6	91LD phenolic resin with carbon cloth CCA-1 and silicon carbide whiskers as reinforcement
473-1a	PPS(5.4)NS-35-C	1.48	60-65	38.4	61.6	14.4	Polyphenylene sulfide QX4375.4(822.6A) resin with sodium sulfide curing agent and carbon cloth as reinforcement.
473-1b	PPS(5.4)PT-35-C	1.32	–	34.7	65.3	–	Polyphenylene sulfide QX4375.4(822.6A) resin with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents and carbon cloth CCA-1 as reinforcement.
473-2a	PPS(5.4)NS-35-C	1.44	–	33.5	66.5	24.2	Polyphenylene sulfide QX4375.4(822-6A) resin with sodium sulfide curing agent and carbon cloth CCA-1 as reinforcement.
473-2b	PPS(5.4)PT-35-C	1.37	–	35.6	64.4	–	Polyphenylene sulfide QX4375.4(822-6A) resin with p-toluenesulfonic acid monohydrate and xylylene gylcol curing agents and carbon cloth CCA-1 as reinforcement.
478	D-PAB-25-R	1.42	–	21.0 (DEN 438) 15.7 (PAB)	63.3	–	DEN 438 epoxy novolac resin with polyamino-borane as filler and Refrasil cloth C100-48 high silica content fabric as reinforcement
479	D-35-AS84	1.46	70	34.6	65.4	–	DEN 438 epoxy novolac resin with Astrosil 84 high silica content cloth as reinforcement
480	D-35-AS41B	1.61	60	35.1	64.9	–	DEN 438 epoxy novolac resin with Astrosil 11341B high silica content cloth as reinforcement
481-1	9-FP-30-GU	1.29	0	33.1 $(91LD)_1$ 14.3 (PFP)	52.6	–	91LD phenolic resin with poly(perfluoro-phenylene) as filler and graphite cloth G1550, uncoated, as reinforcement
481-2		1.30	15	28.6 $(91LD)_1$ 15.1 (PFP)	56.3	–	
481-3		1.31	15	33.2 $(91LD)_1$ 15.9 (PFP)	50.9	–	

TABLE I (CONTINUED)

PROPERTIES OF PELLET SPECIMENS

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent			Description of Material
				Resin	Reinforcement	Volume-Percent Voids in Resin	
482	PBIC-35-R	1.21	–	35.4_2	64.6_2	–	PBI-carborane resin with Refrasil cloth C 100-48 high silica content fabric as reinforcement
483a-1	9-PBIC-30-GU	1.30	24	32.9 (91LD) 13.9 (PBI-C)	53.2	–	91LD phenolic resin with PBI-carborane resin as filler and graphite cloth G1550, uncoated, as reinforcement
483a-2		1.31	24	32.4 (91LD) 14.5 (PBI-C)	53.1	–	
483b-3		1.27	25	31.3 (91LD) 14.8 (PBI-C)	53.9	–	
484	9-BBB-30-GU	–	– –	31.6 (91LD) 15.3 (BBB polymer)	53.1	–	91LD phenolic resin with bisbenzimidazobenzophenanthroline as filler and graphite cloth G1550, uncoated, as reinforcement
485	DPPX-35-GU	1.22	–	41.7_2	58.3_2	–	Poly (α,α' diphenyl-p-xylylidene) resin with graphite cloth G1550, uncoated, as reinforcement.
486	DPMX-35-GU	1.32	–	30.6_2	69.4_2	–	Poly (α,α' diphenyl-m-xylylidene) resin with graphite cloth G1550, uncoated, as reinforcement.
518	PP412B-35-C	1.34	55	36.5	63.5	30.5	Abchar 412B polyphenylene resin with carbon cloth CCA-1 as reinforcement
520	PP413-PP700-30-C	1.31	41	29.7 (Abchar 413) 15.1 (Abchar 700)	55.2	34.9	Abchar 413 polyphenylene resin with Abchar 700 polyphenylene as filler and carbon cloth CCA-1 as reinforcement
521	PP413-35-CLA	1.28	65	36.4	63.6	36.2	Abchar 413 polyphenylene resin with carbon cloth CCA-1, low alkalinity (SS1641) as reinforcement
522	PP412B-PP700-30-C	1.34	53	30.3 (Abchar 412B) 14.9 (Abchar 700)	54.8	30.9	Abchar 412B polyphenylene resin with Abchar 700 polyphenylene as filler and carbon cloth CCA-1 as reinforcement

TABLE I (CONTINUED)

PROPERTIES OF PELLET SPECIMENS

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent: Resin	Composition, Weight-Percent: Reinforcement	Composition, Weight-Percent: Volume-Percent Voids in Resin	Description of Material
524-1 524-2	T-BNF	1.94 2.06	–		See Note 3 See Note 3	–	Teflon 30 resin with boron nitride fibers as reinforcement
526-1 526-2	T-100	2.23 2.26	–	100 100	– –	– –	Teflon 30 resin
530a-1 530a-2 530b-3 530c-4	PPL913-35-C	1.29 1.23 1.18 1.26	–	30.0 29.1 32.8 37.1	70.0 70.9 67.2 62.9	41.5 47.7 47.7 36.8	Abchar L913 polyphenylene resin with carbon cloth CCA-1 as reinforcement
531	PPL1112-35-C	1.17	–	28.3 [4]	71.7 [4]	53.2 [4]	Abchar L1112 polyphenylene resin with carbon cloth CCA-1 as reinforcement
532a-1 532b-2 532c-3	PPH913-35-CT	1.34 1.33 1.40	–	41.6 [5] 54.8 [5] 31.4 [5]	58.4 45.2 68.6	– – –	Abchar H913 polyphenylene resin with pre-treated carbon cloth CCA-1 as reinforcement
533a-1 533b-2 533c-3	PPH1013-35-CT	1.33 1.33 1.30	–	35.0 [5] 34.8 [5] 34.0 [5]	65.0 65.2 66.0	– – –	Abchar H1013 polyphenylene resin with pre-treated carbon cloth CCA-1 as reinforcement
537	PY1-35-C	0.97	–	13.0	87.0	82.3	Polyimidazopyrrolone (PMDA-DAB) resin (Pyrrone) with carbon cloth CCA-1 as reinforcement
539a-1 539a-2 539b-3	SC1-35-T25	1.32 1.32 1.32	51 52 50	37.1 37.2 37.4	62.9 62.8 62.6	–	SC1008 phenolic resin with Thornel 25 graphite fiber tape as reinforcement
541/552	SC1-35-WYB85	1.23	67	37.0	63.0	–	SC1008 phenolic resin with graphite yarn WYB 85 1/2 fiber tape as reinforcement
543/553	SC1-35-VYB70	1.40	65	34.0	66.0	–	SC1008 phenolic resin with carbon yarn VYB 70 1/2 fiber tape as reinforcement
543b	SC1-35-VYB70	1.41	–	36.1	63.9	–	SC1008 phenolic resin with carbon yarn VYB 70 1/2 fiber tape as reinforcement
545-1 545-2 545-3	SK703-35-T25	1.14 1.21 1.22	– – –	15.2 24.4 24.2	84.8 75.6 75.8	– – –	Skybond 703 polyimide resin with Thornel 25 graphite fiber tape as reinforcement
547-1 547-2 547-3	DP25-35-T25	1.29 1.29 1.29	35 35 35	36.1 33.6 33.6	63.9 66.4 66.4	– – –	DP-25-10 phenyl aldehyde resin with Thornel 25 graphite fiber tape as reinforcement
548-1 548-2 548-3	SC1-35-QY	1.59 1.59 1.58	80 80 80	36.6 37.3 37.6	63.4 62.7 62.4	– – –	SC1008 phenolic resin with quartz yarn fiber tape as reinforcement
549-1 549-2 549-3	PPP-35-T25	1.31 1.31 1.31	20 20 20	31.2 33.3 33.6	68.8 66.7 66.4	– – –	p-Phenylphenol phenol formaldehyde resin with Thornel 25 graphite fiber tape as reinforcement

TABLE I (CONTINUED)

PROPERTIES OF PELLET SPECIMENS

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent: Resin	Composition, Weight-Percent: Reinforcement	Composition, Weight-Percent: Volume-Percent Voids in Resin	Description of Material
550-1 550-2	PP413-35-T25	1.17 1.09	10 10	35.0 37.3	65.0 62.7	– –	Abchar 413 polyphenylene resin with Thornel 25 graphite fiber tape as reinforcement
551-1 551-2 551-3	DP4-35-T25	1.31 1.31 1.31	25 25 35	34.5 35.5 34.7	65.5 64.5 65.3	– – –	DP-4-31 phenyl aldehyde resin with Thornel 25 graphite fiber tape as reinforcement
555	PPSP-35-C	1.37	–	37.2	62.8	–	Polyphenylene sulfide resin (Phillips) with carbon cloth CCA-1 as reinforcement
556	9-FP-30-C	1.41	–	31.1 (91LD) 14.7 (PFP)	54.2	–	91LD phenolic resin with poly(perfluoro-phenylene) as filler and carbon cloth CCA-1 as reinforcement
559	9-BBB-30-C	1.37	65	34.1 (91LD) 14.1 (BBB poly-mer)	51.8	–	91LD phenolic resin with bisbenzimidazo-benzophenanthroline polymer as a filler and carbon cloth CCA-1 as reinforcement
560-1 560-2	9-35-T40F	1.35 1.35	35 35	34.8 34.6	65.2 65.4	– –	91LD phenolic resin with Thornel 40 graphite fabric as reinforcement
561	PP412B-35-C	1.34	–	34.0	66.0	32.8	Abchar 412B polyphenylene resin with carbon cloth CCA-1 as reinforcement

[1] Resin content after molding and prior to postcure.

[2] As molded. Specimens not postcured.

[3] Received from AFML. Composition weight-percent not reported.

[4] Values are approximate.

[5] Combined weight-percent of resin and cloth pretreatment.

TABLE II

PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent			Description of Material
				Resin	Reinforcement	Volume-Percent Voids in Resin	
407	F170-40-C	1.25	–	40.2	59.8	–	F170 polyimide resin with carbon cloth CCA-1 as reinforcement
408	F171-40-C	1.36	–	35.4	64.6	–	F171 polyarylene-phenolic resin with carbon cloth CCA-1 as reinforcement
409	F172-40-C	1.37	74	41.6	58.4	–	F172 polyphenylene-phenolic resin with carbon cloth CCA-1 as reinforcement
467-1 467-2	CP-35-C	1.32 1.31	75 76	33.6 33.4	66.4 66.6	– –	Chrome-P metal organic phenolic resin with carbon cloth CCA-1 as reinforcement
468-1a-1 468-1a-2	PPS(5.4)NS-35-C	1.54 1.55	60-65 60-65	34.8 36.2	65.2 63.8	10.6 8.3	Polyphenylene sulfide QX4375.4(822-6A) resin with sodium sulfide curing agent and carbon cloth CCA-1 as reinforcement
468-1b-1 468-1b-2	PPS(5.4)PT-35-C	1.44 1.40	– –	34.9 36.0	65.1 64.0	–	Polyphenylene sulfide QX4375.4(822-6A) resin with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents and carbon cloth CCA-1 as reinforcement
469	PPS(5.4)PT-35-R	1.59	–	32.8	67.2	–	Polyphenylene sulfide QX4375.4(822-6A) resin with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents and Refrasil C100-48 high silica content cloth.
491-1 491-2	TP(H)-35-C	1.49 1.48	70 68	36.2 35.2	63.8 64.8	18.2 20.4	High tungsten-P metal organic phenolic resin with carbon cloth CCA-1 as reinforcement
534-1 534-2 534-3 534-4	9-35-C	1.44 1.32 1.44 1.42	70 68 70 70	37.6 36.4 36.9 36.7	62.4 63.6 63.1 63.3	15.9 30.2 10.7 19.0	91LD phenolic resin with carbon cloth CCA-1 as reinforcement
535a-1 535a-2 535a-3 535a-4 535b-5 535b-6	9-35-R	1.67 1.67 1.69 1.67 1.65 1.67	70 65 65 67 65 65	34.4 34.6 34.4 34.0 33.9 33.6	65.6 65.4 65.6 66.0 66.1 66.4	4.8 4.8 3.2 4.8 7.9 5.6	91LD phenolic resin with Refrasil cloth C100-48 high silica content fabric as reinforcement

TABLE II (CONTINUED)

PROPERTIES OF ROCKET NOZZLES

Data Sheet Number	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent			Description of Material
				Resin	Reinforcement	Volume-Percent Voids in Resin	
536-1 536-2	PY1-35-C	0.95 0.88	– –	20.4 29.8	79.6 70.2	74.6 70.0	Polyimidazopyrrolone (PMDA-DAB) resin (Pyrrone) with carbon cloth CCA-1 as reinforcement
538-1 538-2	SC1-35-T25	1.36 1.26	55 53	32.5 34.3	67.5 65.7	– –	SC1008 phenolic resin with Thornel 25 graphite fiber tape as reinforcement
540/552	SC1-35-WYB85	1.40	67	37.0	63.0	–	SC1008 phenolic resin with graphite yarn WYB 85 1/2 fiber tape as reinforcement
542/553	SC1-35-VYB70	1.37	65	34.0	66.0	–	SC1008 phenolic resin with carbon yarn VYB 70 1/2 fiber tape as reinforcement
542b	SC1-35-VYB70	1.40	65	36.0	64.0	–	SC1008 phenolic resin with carbon yarn VYB 70 1/2 as fiber tape reinforcement
546-1 546-2	DP25-35-T25	1.31 1.29	47 47	37.3 37.3	62.7 62.7	– –	DP-25-10 phenyl aldehyde resin with Thornel 25 graphite fiber tape as reinforcement
562-1	9-FP-30-C	1.42	–	31.8 (91LD) 14.6 (PFP)	53.6	–	91LD phenolic resin with poly(perfluorophenylene) as filler and carbon cloth CCA-1 as reinforcement
562-2		1.45	–	31.6 (91LD) 14.7 (PFP)	53.7	–	
565-1	9-BBB-30-C	1.41	72	33.1 (91LD) 14.3 (BBB polymer)	52.6	–	91LD phenolic resin with bisbenzimidazobenzophenanthroline polymer as filler and carbon cloth CCA-1 as reinforcement
565-2		1.42	75	32.3 (91LD) 14.5 (BBB polymer)	53.2	–	
566	9-35-T40F	1.39	45	35.9	64.1	–	91LD phenolic resin with Thornel 40 graphite fabric as reinforcement
567	PP412B-35-C	1.38	–	34.2	65.8	29.0	Abchar 412B polyphenylene resin with carbon cloth CCA-1 as reinforcement
571-1 571-2	9-35-C	1.45 1.45	78 78	36.9 36.8	63.1 63.2	15.9 15.9	91LD phenolic resin with carbon cloth CCA-1 as reinforcement

TABLE III

PROPERTIES OF MISCELLANEOUS TYPES OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Density, gm/cc	Barcol Hardness	Composition, Weight-Percent: Resin	Reinforcement	Voids in Resin	Description of Material
462	Laminated Square	F171-40-C	1.34	–	27.7	72.3	–	F171 polyarylene-phenolic resin with carbon cloth CCA-1 as reinforcement
463	Laminated Square	F172-40-C	1.38	–	38.9	61.1	–	F172 polyphenylene-phenolic resin with carbon cloth CCA-1 as reinforcement
525-1 525-2	Cylinder	T-100	2.26 2.24	– –	100 100	– –	– –	Teflon 30 resin
527	Laminate	PP412-40-AQ570	1.55	40	39.6	60.4	14.6	Abchar 412 polyphenylene resin with Astroquartz 570 high silica content fabric as reinforcement
529-1 529-2	Cylinder	PP412-40-AQ570	1.59 1.60	– –	40.3 40.1	59.7 59.9	10.0 9.2	Abchar 412 polyphenylene resin with Astroquartz 570 high silica content fabric as reinforcement
554	Molding	PP413-35-AQ570	1.55	35-50	36.4	63.6	20.0	Abchar 413 polyphenylene resin with Astroquartz 570 high silica content fabric as reinforcement
572a-1 572a-2 572a-3	TGA		–	–	100	–	–	Polyphenylene sulfide resin (Phillips) Polyphenylene resin (Abchar L913) Polyphenylene resin (Abchar 412B)
572b-4	TGA		–	–	100	–	–	91LD phenolic resin
572b-5			–	–	66 2/3 (91LD) 33 1/3 (PFP)	–	–	91LD phenolic resin with poly(perfluorophenylene) as filler
572b-6			–	–	66 2/3 (91LD) 33 1/3 (BBB Polymer)	–	–	91LD phenolic resin with bisbenzimidazobenzophenanthroline polymer as filler

TABLE IV

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Material Code	Date Requested (AFML Letter Reference or Telecon)	Date of Shipment	Detailed Letter Report: Reference Number	Detailed Letter Report: Date
407	Nozzle	F170-40-C	11 Aug 65, Appendix D (1k)	15 Feb 68	2748. 1/1267	6 Mar 68
408	Nozzle	F171-40-C	11 Aug 65, Appendix D (1l)	15 Feb 68	2748. 1/1267	6 Mar 68
409	Nozzle	F172-40-C	11 Aug 65, Appendix D (1m)	15 Feb 68	2748. 1/1267	6 Mar 68
440	Pellet	9-28-SCWC	11 Aug 65, Appendix A (1d)	25 May 67	2748. 1/1129	21 Jul 67
462	Laminated square	F171-40-C	11 Aug 65, Appendix C (1b)	15 Feb 68	2748. 1/1267	6 Mar 68
463	Laminated square	F172-40-C	11 Aug 65, Appendix C (1c)	15 Feb 68	2748. 1/1267	6 Mar 68
467	Nozzle	CP-35-C	1 Feb 66, Appendix B (1c)	30 Nov 67	2748. 1/1264	27 Feb 68
468-1a	Nozzle	PPS(5. 4)NS-35-C	1 Feb 66, Appendix B (1d) and Telecon 25 Sept 67	3 Nov 67	2748. 1/1264	27 Feb 68
468-1b	Nozzle	PPS(5. 4)PT-35-C	1 Feb 66, Appendix B (1c) and Telecon 25 Sept 67	30 Nov 67	2748. 1/1264	27 Feb 68
469	Nozzle	PPS(5. 4)PT-35-R	1 Feb 66, Appendix B (1e) and Telecon 25 Sept 67	5 Jan 68	2748. 1/1264	27 Feb 68
473-1a	Pellet	PPS(5. 4)NS-35-C	1 Feb 66, Appendix A (3a) and Telecon 25 Sept 67	3 Nov 67	2748. 1/1264	27 Feb 68
473-1b	Pellet	PPS(5. 4)PT-35-C	1 Feb 66, Appendix A (3a) and Telecon 25 Sept 67	30 Nov 67	2748. 1/1264	27 Feb 68
473-2a	Pellet	PPS(5. 4)NS-35-C	1 Feb 66, Appendix A (3a) and Telecon 25 Sept 67	5 Jan 68	2748. 1/1264	27 Feb 68
473-2b	Pellet	PPS(5. 4)PT-35-C	1 Feb 66, Appendix A (3a) and Telecon 25 Sept 67	5 Jan 68	2748. 1/1264	27 Feb 68
478	Pellet	D-PAB-25-R	21 Apr 66, Appendix B (1d)	30 Mar 67	2748. 1/1069	12 Apr 67
479	Pellet	D-35-AS84	21 Apr 66, Appendix B (1e)	8 Mar 67	2748. 1/1069	12 Apr 67
480	Pellet	D-35-AS41B	21 Apr 66, Appendix B (1f)	13 Feb 67	2748. 1/1069	12 Apr 67
481	Pellet	9-FP-30-GU	21 Apr 66, Appendix B (1g)	13 Feb 67	2748. 1/1069	12 Apr 67
482	Pellet	PBIC-35-R	21 Apr 66, Appendix B (1h)	25 May 67	2748. 1/1129	21 Jul 67
483a & b	Pellet	9-PBIC-30-GU	21 Apr 66, Appendix B (1i) and Telecon 1 Mar 67	30 Nov 67	2748. 1/1264	27 Feb 68
484	Pellet	9-BBB-30-GU	21 Apr 66, Appendix B (1j)	30 Mar 67	2748. 1/1069	12 Apr 67
485	Pellet	DPPX-35-GU	21 Apr 66, Appendix B (1k)	25 May 67	2748. 1/1129	21 Jul 67
486	Pellet	DPMX-35-GU	21 Apr 66, Appendix B (1l)	25 May 67	2748. 1/1129	21 Jul 67
491	Nozzle	TP(H)-35-C	21 Apr 66, Appendix D (1d)	20 Oct 67	2748. 1/1264	27 Feb 28
518	Pellet	PP412B-35-C	19 Oct 66, Appendix E (1a)	13 Feb 67	2748. 1/1069	12 Apr 67
520	Pellet	PP413-PP700-30-C	19 Oct 66, Appendix E (1c)	13 Feb 67	2748. 1/1069	12 Apr 67
521	Pellet	PP413-35-CLA	19 Oct 66, Appendix E (1d)	13 Feb 67	2748. 1/1069	12 Apr 67
522	Pellet	PP412B-PP700-30-C	19 Oct 66, Appendix E (1e)	13 Feb 67	2748. 1/1069	12 Apr 67
524	Pellet	T-BNF	18 Nov 66, Appendix B (1c)	13 Apr 67	2748. 1/1069	12 Apr 67
525	Cylinder	T-100	18 Nov 66, Appendix B (2a & 2b)	13 Apr 67	2748. 1/1069	12 Apr 67
526	Pellet	T-100	18 Nov 66, Appendix B (2c)	13 Apr 67	2748. 1/1069	12 Apr 67
527	Laminate	PP412-40-AO570	18 Nov 66, Appendix B (3)	13 Apr 67	2748. 1/1069	12 Apr 67
529	Cylinder	PP412-40-AQ570	18 Nov 66, Appendix B (5a & 5b)	13 Apr 67	2748. 1/1069	12 Apr 67
530a, b & c	Pellet	PPL913-35-C	Telecon 11 Jan 67	30 Mar 67	2748. 1/1069	12 Apr 67
531	Pellet	PPL1112-35-C	Telecon 11 Jan 67	26 Jun 67	2748. 1/1129	21 Jul 67

TABLE IV (CONTINUED)

TEST SPECIMEN RECORD

Data Sheet Number	Type of Specimen	Material Code	Date Requested (AFML Letter Reference or Telecon)	Date of Shipment	Detailed Letter Report: Reference Number	Detailed Letter Report: Date
532a, b, & c	Pellet	PPH913-35-CT	Telecon 11 Jan 67	30 Mar 67	2748. 1/1069	12 Apr 67
533a, b, & c	Pellet	PPH1015-35-CT	Telecon 11 Jan 67	30 Mar 67	2748. 1/1069	12 Apr 67
534	Nozzle	9-35-C	Telecon 24 Jan 67	15 Feb 67	2748. 1/1069	12 Apr 67
535a & b	Nozzle	9-35-R	Telecon 24 Jan 67	15 Feb 67	2748. 1/1069	12 Apr 67
536	Nozzle	PY1-35-C	Telecon 18 Jan 67	8 Mar 67	2748. 1/1069	12 Apr 67
537	Pellet	PY1-35-C	Telecon 18 Jan 67	8 Mar 67	2748. 1/1069	12 Apr 67
538	Nozzle	SC1-35-T25	19 Oct 66, Appendix D (1a)	20 Oct 67	2748. 1/1264	27 Feb 68
539a & b	Pellet	SC1-35-T25	19 Oct 66, Appendix D (1a)	25 May 67	2748. 1/1129	21 Jul 67
540/552	Nozzle	SC1-35-WYB85	19 Oct 66, Appendix D (1b)	13 Jun 67	2748. 1/1129	21 Jul 67
541/552	Pellet	SC1-35-WYB85	19 Oct 66, Appendix D (1b)	13 Jun 67	2748. 1/1129	21 Jul 67
542/553	Nozzle	SC1-35-VYB70	19 Oct 66, Appendix D (1c)	13 Jun 67	2748. 1/1129	21 Jul 67
542b	Nozzle	SC1-35-VYB70	19 Oct 66, Appendix D (1c)	28 Jul 67	2748. 1/1264	27 Feb 68
543/553	Pellet	SC1-35-VYB70	19 Oct 66, Appendix D (1c)	13 Jun 67	2748. 1/1129	21 Jul 67
543b	Pellet	SC1-35-VYB70	19 Oct 66, Appendix D (1c)	26 Jun 67	2748. 1/1129	21 Jul 67
545	Pellet	SK703-35-T25	19 Oct 66, Appendix D (1d)	28 Jul 67	2748. 1/1264	27 Feb 68
546	Nozzle	DP25-35-T25	19 Oct 66, Appendix D (1e)	28 Jul 67	2748. 1/1264	27 Feb 68
547	Pellet	DP25-35-T25	19 Oct 66, Appendix D (1e)	20 Oct 67	2748. 1/1264	27 Feb 68
548	Pellet	SC1-35-QY	19 Oct 66, Appendix D (2a)	26 Jun 67	2748. 1/1129	21 Jul 67
549	Pellet	PPP-35-T25	19 Oct 66, Appendix D (2b)	20 Oct 67	2748. 1/1264	27 Feb 68
550	Pellet	PP413-35-T25	19 Oct 66, Appendix D (2c)	26 Jun 67	2748. 1/1129	21 Jul 67
551	Pellet	DP4-35-T25	19 Oct 66, Appendix D (2d)	28 Jul 67	2748. 1/1264	27 Feb 68
554	Molding	PP413-35-AQ570	Telecon 25 Jul 67	2 Aug 67	2748. 1/1264	27 Feb 68
555	Pellet	PPSP-35-C	5 Oct 67, Appendix B (1a)	15 Feb 68	2748. 1/1267	6 Mar 68
556	Pellet	9-FP-30-C	5 Oct 67, Appendix B (1b)	15 Feb 68	2748. 1/1267	6 Mar 68
559	Pellet	9-BBB-30-C	5 Oct 67, Appendix B (1e)	5 Jan 68	2748. 1/1264	27 Feb 68
560	Pellet	9-35-T40F	5 Oct 67, Appendix B (1f)	30 Nov 67	2748. 1/1264	27 Feb 68
561	Pellet	PP412B-35-C	5 Oct 67, Appendix B (1g)	15 Feb 68	2748. 1/1267	6 Mar 68
562	Nozzle	9-FP-30-C	5 Oct 67, Appendix B (2a)	30 Nov 67	2748. 1/1264	27 Feb 68
565	Nozzle	9-BBB-30-C	5 Oct 67, Appendix B (2d)	5 Jan 68	2748. 1/1264	27 Feb 68
566	Nozzle	9-35-T40F	5 Oct 67, Appendix B (2e)	30 Nov 67	2748. 1/1264	27 Feb 68
567	Nozzle	PP412B-35-C	5 Oct 67, Appendix B (2f)	15 Feb 68	2748. 1/1267	6 Mar 68
571	Nozzle	9-35-C	Telecon 2 Feb 68	15 Feb 68	2748. 1/1267	6 Mar 68
572a & b	TGA	–	5 Oct 67, Appendix B (4a thru 4f)	15 Feb 68	2748. 1/1267	6 Mar 68

TABLE V

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin	Data Sheet Number	Type Specimen
Astroquartz 570 (high silica content fabric)	Abchar 412	527 529	Laminate Cylinder
	Abchar 413	554	Molding
Astrosil 84 (high silica content fabric)	DEN 438	479	Pellet
Astrosil 11341B (high silica content fabric)	DEN 438	480	Pellet
Boron nitride fibers	Teflon 30	524	Pellet
Carbon cloth CCA-1	91 LD	534 571	Nozzle Nozzle
	91 LD with BBB polymer as filler	559 565	Pellet Nozzle
	91 LD with poly(perfluorophenylene) as filler	556 562	Pellet Nozzle
	Abchar 412B	518 561 567	Pellet Pellet Nozzle
	Abchar 412B with Abchar 700 as filler	522	Pellet
	Abchar 413 with Abchar 700 as filler	520	Pellet
	Abchar H913	532 a, b & c	Pellet
	Abchar L913	530 a, b & c	Pellet
	Abchar H1013	533 a, b & c	Pellet
	Abchar L1112	531	Pellet
	Chrome-P	467	Nozzle
Carbon cloth CCA-1	F170	407	Nozzle
	F171	408 462	Nozzle Lam. Sq.
	F172	409 463	Nozzle Lam. Sq.
	Polyimidazopyrrolone (Pyrrone)	536 537	Nozzle Pellet
	Polyphenylene sulfide (Dow) with sodium sulfide curing agent	468-1a 473-1a 473-2a	Nozzle Pellet Pellet
	Polyphenylene sulfide (Dow) with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents	468-1b 473-1b 473-2b	Nozzle Pellet Pellet
	Polyphenylene sulfide (Phillips)	555	Pellet
	Tungsten-P (high)	491	Nozzle
Carbon cloth CCA-1 (low alkalinity-SS1641)	Abchar 413	521	Pellet
Carbon cloth CCA-1 with silicon carbide whiskers	91 LD	440	Pellet
Carbon yarn VYB 70 1/2	SC1008	542/553 542b 543/553 543b	Nozzle Nozzle Pellet Pellet
Graphite cloth G1550, uncoated	91 LD with BBB polymer as filler	484	Pellet
	91 LD with poly(perfluorophenylene) as filler	481	Pellet
	91 LD with PBI-carborane as filler	483 a&b	Pellet
	Poly (o, o'-diphenyl-m-xylylidine)	486	Pellet
	Poly (o, o'-diphenyl-p-xylylidine)	485	Pellet

TABLE V (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF REINFORCEMENT

Type of Reinforcement	Type of Resin	Data Sheet Number	Type Specimen
Graphite yarn WYB85 1/2	SC1008	540/552 541/552	Nozzle Pellet
Quartz yarn	SC1008	548	Pellet
Refrasil cloth C100-48 (high silica content fabric)	91LD	535 a&b	Nozzle
	DEN 438 with polyaminoborane as filler	478	Pellet
	PBI-carborane	482	Pellet
	Polyphenylene sulfide (Dow) with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents	469	Nozzle
Thornel 25 graphite fibers	Abchar 413	550	Pellet
	DP4-31	551	Pellet
	DP25-10	546 547	Nozzle Pellet
	p-Phenylphenol phenol formaldehyde	549	Pellet
	SC1008	538 539 a&b	Nozzle Pellet
	Skybond 703	545	Pellet
Thornel 40 graphite fabric	91LD	560 566	Pellet Nozzle

TABLE VI

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
91LD	Phenolic	None	572b	TGA
		Carbon cloth CCA-1	534 571	Nozzle Nozzle
		Carbon cloth CCA-1 and silicon carbide whiskers	440	Pellet
		Refrasil cloth C100-48	535 a&b	Nozzle
		Thornel 40 graphite fabric	560 566	Pellet Nozzle
91LD with BBB polymer as filler	Phenolic with bizbenzimidazo-benzophenanthroline filler	None	572b	TGA
		Carbon cloth CCA-1	559 565	Pellet Nozzle
		Graphite cloth G1550, uncoated	484	Pellet
91LD with PBI-carborane as filler	Phenolic with polybenzimidazole-carborane filler	Graphite cloth G1550, uncoated	483 a&b	Pellet
91LD with poly-(perfluoro-phenylene) as filler	Phenolic with poly(perfluoro-phenylene) as filler	None	572b	TGA
		Carbon cloth CCA-1	556 562	Pellet Nozzle
		Graphite cloth G1550, uncoated	481	Pellet
Abchar 412	m-polyphenylene	Astroquartz 570 cloth	527 529	Laminate Cylinder
Abchar 412B	m-polyphenylene	None	572a	TGA
		Carbon cloth CCA-1	518 561 567	Pellet Pellet Nozzle
Abchar 412B with Abchar 700 as filler	m-polyphenylene with a poly-phenylene filler	Carbon cloth CCA-1	522	Pellet

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
Abchar 413	m-polyphenylene	Astroquartz 570 cloth	554	Molding
		Carbon cloth CCA-1 (low alkalinity-SS1641)	521	Pellet
		Thornel 25 graphite fibers	550	Pellet
Abchar 413 with Abchar 700 as filler	m-polyphenylene with a poly-phenylene filler	Carbon cloth CCA-1	520	Pellet
Abchar H913	High molecular weight m-polyphenylene	Carbon cloth CCA-1	532 a, b&c	Pellet
Abchar L913	Low molecular weight m-polyphenylene	None	572a	TGA
		Carbon cloth CCA-1	530a, b&c	Pellet
Abchar H1013	High molecular weight m-polyphenylene	Carbon cloth CCA-1	533 a, b&c	Pellet
Abchar L1112	Low molecular weight m-polyphenylene	Carbon cloth CCA-1	531	Pellet
Chrome-P	Chrome based metal organic phenolic	Carbon cloth CCA-1	467	Nozzle
DEN 438	Epoxy novolac	Astrosil 84 cloth	479	Pellet
		Astrosil 11341B cloth	480	Pellet
DEN 438 with PAB as filler	Epoxy novolac with polyaminoborane as filler	Refrasil cloth C100-48	478	Pellet
DP 4-31	Phenyl aldehyde	Thornel 25 graphite fibers	551	Pellet

TABLE VI (CONTINUED)

TEST SPECIMENS LISTED ACCORDING TO TYPE OF RESIN

Trade Name or Designation	Type of Resin	Type of Reinforcement	Data Sheet Number	Type Specimen
DP 25-10	Phenyl aldehyde	Thornel 25 graphite fibers	546 547	Nozzle Pellet
F170	Polyimide	Carbon cloth CCA-1	407	Nozzle
F171	Polyarylene-phenolic	Carbon cloth CCA-1	408 462	Nozzle Lam. Sq.
F172	Polyphenylene-phenolic	Carbon cloth CCA-1	409 463	Nozzle Lam. Sq.
PBI-carborane	Polybenzimidazole-carborane	Refrasil cloth C100-48	482	Pellet
p-Phenylphenol phenol formaldehyde	Modified phenolic	Thornel 25 graphite fibers	4[illegible]	Pellet
Poly(α,α'-diphenyl-m-xylylidene)	—	Graphite cloth G1550, uncoated	486	Pellet
Poly(α,α'-diphenyl-p-xylylidine)	—	Graphite cloth G1550, uncoated	485	Pellet
Polyphenylene sulfide (Dow)	Polyphenylene sulfide with sodium sulfide curing agent	Carbon cloth CCA-1	468-1a 473-1a 473-2a	Nozzle Pellet Pellet
Polyphenylene sulfide (Dow)	Polyphenylene sulfide with p-toluenesulfonic acid monohydrate and xylylene glycol curing agents	Carbon cloth CCA-1	468-1b 473-1b 473-2b	Nozzle Pellet Pellet
		Refrasil cloth C100-48	469	Nozzle
Polyphenylene sulfide (Phillips)	Polyphenylene sulfide	None	572a	TGA
		Carbon cloth CCA-1	555	Pellet
Pyrrone	Polymidazopyrrolone (PMDA-DAB)	Carbon cloth CCA-1	536 537	Nozzle Pellet
SC1008	Phenolic	Quartz yarn	548	Pellet
		Thornel 25 graphite fibers	538	Nozzle
			539 a&b	Pellet
SC1008	Phenolic	Carbon yarn VYB70 1/2	542/553 542b 543/553 543b	Nozzle Nozzle Pellet Pellet
		Graphite yarn WYB 85 1/2	540/552 541/552	Nozzle Pellet
Skybond 703	Polyimide	Thornel 25 graphite fibers	545	Pellet
Teflon 30	Polytetrafluoroethylene	None	525 526	Cylinder Pellet
Teflon 30 with boron nitride fibers	Polytetrafluoroethylene with boron nitride fibers	None	524	Pellet
Tungsten-P (high tungsten content)	Tungsten based metal organic phenolic	Carbon cloth CCA-1	491	Nozzle

TABLE VII

FABRICATION DETAILS – PELLET SPECIMENS

Data Sheet Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			+Postcure
					Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time, Minutes	
440	3/4" dia x 5/8" disc	9-28-SCWC	1/8.7 acetone for whiskers 1/6.3 acetone for cloth	Soaking for whiskers Spatula coating for cloth	60 60	60 min at 160°F 60 min at 160°F	20 min at 225°F 20 min at 225°F	300	3300	120	B-1
473-1a	2" dia x 5/8" disc	PPS(5.4)NS-35-C	Not applicable	Dry powder layup	—	—	—	600	3300	120	Extended press cure in lieu of postcure
473-1b	2" dia x 5/8" disc	PPS(5.4)PT-35-C	Not applicable	Dry powder layup	—	—	—	500	3300	960	Extended press cure in lieu of postcure
473-2a	2" dia x 5/8" disc	PPS(5.4)NS-35-C	Not applicable	Dry powder layup	—	—	—	600	3300	1020	Extended press cure in lieu of postcure
473-2b	2" dia x 5/8" disc	PPS(5.4)PT-35-C	Not applicable	Dry powder layup	—	—	—	500	3300	1020	Extended press cure in lieu of postcure
478	2" dia x 5/8" disc	D-PAB-25-R	Thinned with acetone to coating consistency	Spatula coating	60	30 min at 160°F	30 min at 225°F	275	3300	60	See Note 1
479	2" dia x 5/8" disc	D-35-AS84	1/1 acetone	Spatula coating	60	30 min at 160°F	20 min at 225°F	See Note 2	3300	See Note 2	See Note 2
480	2" dia x 5/8" disc	D-35-AS41B	1/1 acetone	Brush coating	60	30 min at 160°F	40 min at 225°F 20 min at 250°F	See Note 2	3300	See Note 2	See Note 2
481-1 481-2 481-3	3/4" dia x 5/8" disc	9-FP-30-GU	1/0.5 acetone	Brush coating	60	60 min at 160°F	30 min at 225°F	300	3300	120	B-1
482	2" dia x 5/8" disc	PBIC-35-R	1/2.5 NMP	Dip coating	—	30 min at 300°F + 8 hrs at 300°F under vacuum	15 min at 400°F	700	5000	120	None

TABLE VII (CONTINUED)

FABRICATION DETAILS – PELLET SPECIMENS

Data Sheet Number	Dimensions of Original Laminate or Molding	* Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			+ Postcure
					Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time, Minutes	
483a-1 483a-2 483b-3	3/4" dia x 5/8" disc	9-PBIC-30-GU	—	Dip coating	60	45 min at 180°F	—	300	1000	120	B-1
484	2" dia x 5/8" disc	9-BBB-30-GU	1/1 acetone	Spatula coating	—	60 min at 160°F	30 min at 225°F	300	3300	120	B-1
485	3/4" dia x 5/8 disc	DPPX-35-GU	1/5 NMP	Spatula coating	—	90 min at 300°F	30 min at 400°F	650	500	120	None
486	3/4" dia x 5/8 disc	DPMX-35-GU	1/5 NMP	Spatula coating	—	40 min at 300°F	30 min at 400°F	650	500	120	None
518	3-1/2" dia x 5/8" disc	PP412B-35-C	Used as received	Dip coating	30	10 min at 160°F	Vacuum dried at room temp for 17 hrs	400	3300	120	I-3
520	3-1/2" dia x 5/8" disc	PP413-PP700-30-C	Used as received	Spatula coating	30	10 min at 160°F	Vacuum dried at room temp for 18 hrs	400	3300	120	I-3
521	3-1/2" dia x 5/8" disc	PP41?-35-CLA	Used as received	Brush coating	30	15 min at 160°F	Vacuum dried at room temp for 16 hrs	400	3300	120	I-3
522	3-1/2" dia x 5/8" disc	PP412B-PP700-30-C	Used as received	Spatula coating	30	10 min at 160°F	Vacuum dried at room temp for 17 hrs	400	3300	120	I-3
524-1 524-2	1" dia x 5/8 disc	T-3NF	Not applicable	Not applicable	Not appli-cable	Not appli-cable	Not applicable	700	5000	15	None
526-1 526-2	1" dia x 5/8 disc	T-100	Not applicable	Not applicable	Not appli-cable	Not appli-cable	Not applicable	700	5000	15	None
530a-1 530a-2 530b-3 530c-4	1" dia x 1-1/4 cylinder	PPL913-35-C	Used as received	Spatula coating	30	20 min at 160°F	—	400	3300	120	I-4
531	1" dia x 1-1/4 cylinder	PPL1112-35-C	Used as received	Spatula coating	30	25 min at 400°F	—	600	3300	120	I-4

TABLE VII (CONTINUED)

FABRICATION DETAILS – PELLET SPECIMENS

Data Sheet Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			*Postcure
					Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time Minutes	
532a-1 532b-2 532c-3	1" dia x 1-1/4" cylinder	PPH913-35-CT	Resin powder-chloroform slurry	Spatula coating	—	10 min at 160°F	—	500	3300	120	I-4
533a-1 533b-2 533c-3	1" dia x 1-1/4 cylinder	PPH1013-35-CT	Resin powder-chloroform slurry	Spatula coating	—	10 min at 160°F	—	500	3300	120	I-4
537	4" x 2-1/2" x 5/8" Laminate	PY1-35-C	Used as received	Dip coating	10 min between dips	3 min at 200°F after every other dip	12 min at 200°F	300	1000	60	L-1
539a-1 539a-2 539b-3	1" x 1" x 5/8" block	SC1-35-T25	Used as received	Prepared by AFML	—	60 min at 160°F	60 min at 225°F	300	3300	120	B-1
541/552	1-3/4" x 1-3/4" x 3" Block	SC1-35-WYB85	Used as received	Prepared by AFML	—	60 min at 160°F	40 min at 225°F	300	10,000	120	B-1
543/553	1-3/4" x 1-3/4" x 3" Block	SC1-35-VYB70	Used as received	Prepared by AFML	—	60 min at 160°F	60 min at 225°F	300	10,000	120	B-1
543b	1" x 1" x 5/8"	SC1-35-VYB70	Used as received	Prepared by AFML	—	60 min at 160°F	60 min at 225°F	300	3300	120	B-1
545-1 545-2 545-3	1" x 1" x 5/8"	SK703-35-T25	Used as received	Prepared by AFML	—	60 min at 200°F	90 min at 250 F	450	3300	120	E
547-1 547-2 547-3	1" x 1" x 5/8" Block	DP25-35-T25	Used as received	Prepared by AFML	—	60 min at 160°F	30 min at 225°F	300	3300	120	B-1
548-1 548-2 548-3	1" x 1" x 5/8"	SC1-35-QY	Used as received	Prepared by AFML	—	60 min at 160°F	60 min at 225°F	400	3300	120	B-1
549-1 549-2 549-3	1" x 1" x 5/8" Block	PPP-35-T25	Used as received	Prepared by AFML	—	—	10 min at 160°F	300	3300	120	B-1
550-1 550-2	1" x 1" x 5/8"	PP413-35-T25	Used as received	Prepared by AFML	—	35 min at 160°F	—	400	3300	120	I-3
551-1 551-2 551-3	1" x 1" x 5/8" Block	DP4-35-T25	Used as received	Prepared by AFML	—	60 min at 160°F	25 min at 225°F	300	3300	120	B-1

TABLE VII (CONTINUED)

FABRICATION DETAILS – PELLET SPECIMENS

Data Sheet Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			*Postcure
					Air Dry, Minutes	Oven Dry		Temp. °F	Pressure, PSI	Time, Minutes	
555	2" dia x 5/8" disc	PPSP-35-C	Not applicable	Dry powder layup	–	–	–	650	3300	120	I-3[3]
556	2" dia x 5/8" disc	9-FP-30-C	1/2.8 acetone	Spatula coating	60	60 min at 160 F	15 min at 225 F	300	3300	120	B-1
559	2" dia x 5/8" disc	9-BBB-30-C	1/2 acetone	Spatula coating	60	30 min at 160 F	15 min at 225 F	300	1000	120	B-1
560-1 560-2	2" dia x 5/8" disc	9-35-T40F	1/3 acetone	Spatula coating	60	60 min at 160 F	25 min at 225 F	300	3300	120	B-1
561	3-1/2" dia x 5/8" disc	PP412B-35-C	Used as received	Brush coating	20	20 min at 160 F	–	400	3300	120	I-3

[1] Part was placed back in the mold and postcured under 3300 psi pressure as follows: 4 hrs at 300 F, 16 hrs at 350 F, 1 hr. at 400 F, 7 hrs. cooling to below 200°F.

[2] Part was cured over a prolonged period under pressure in lieu of a postcure. The cure cycle was as follows: 18 hrs at 300 F, 24 hrs from 300 to 400°F, 1 hr at 400 F, 7 hrs cooling to room temperature.

[3] Data Sheet 555: Additional hour in mold at 650 F and 3300 psi pressure after standard I-3 postcure. Specimen was cooled under pressure.

* The following postcure schedules were used:

- B-1 — 18 hrs at 275 F, 72 hrs from 275 to 400 F, 4 hrs at 400 F, 7 hrs cooling to below 200 F.
- E — 24 hrs at 375 F, 24 hrs at 435 F, 24 hrs at 475 F, 24 hrs at 575 F, 6 hrs between temperatures. Cool to below 200 F. Specimen No. 1 of Data Sheet No. 545 was postcured an additional 12 hrs at 635 F, 12 hrs at 675 F and 12 hrs at 700 F before cooling.
- I-3 — 18 hrs at 275 F, 108 hrs from 275 to 550 F, 6 hrs at 550 F, 7 hrs cooling to below 200 F. Specimens were postcured in argon.
- I-4 — 18 hrs at 275 F, 120 hrs from 275 to 750 F, 7 hrs cooling to below 200 F. Specimens were postcured in argon.
- L-1 — 165 hrs from 275 to 600 F, 27 hrs at 600 F, 7 hrs cooling to below 200 F.

TABLE VIII

FABRICATION DETAILS — ROCKET NOZZLES

Data Sheet Number	Nozzle Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			+Postcure
						Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time, Minutes	
407	K-65-1	3" x 1-5/8" dia cylinder	F170-40-C	1/2 acetone	Spatula coating	60	60 min at 200°F	30 min at 250°F	400	10,000	120	B-7
408	K-66-1	3" x 1-5/8" dia cylinder	F171-40-C	1/2 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	350	10,000	120	B-7
409	K-67-1	3" x 1-5/8" dia cylinder	F172-40 C	1/1 acetone	Spatula coating	60	60 min at 160°F	30 min at 225°F	300	10,000	120	B-8
467	K-76-1 K-76-2	3" x 1-5/8" dia cylinder	CP-35-C	1/2 acetone	Spatula coating	60	-	450 min at 180°F	180 &250	10,000	45 min contact 120 - (180°F) 300 - (250°F)	J-2
468-1a	K-77-1 K-77-2	3" x 1-5/8" dia cylinder	PPS(5.4)NS-35-C	Not applicable	Dry powder layup	-	-	-	600	10,000	120	Postcured in press for 16 hrs at 600°F & 10,000 psi
468-1b	K-96-1 K-96-2	3" x 1-5/8" dia cylinder	PPS(5.4)PT-35-C	Not applicable	Dry powder layup	-	-	-	500	10,000	1440	Extended press cure in lieu of postcure
469	K-78-1	3" x 1-5/8" dia cylinder	PPS(5.4)PT-35-R	Not applicable	Dry powder layup	-	-	-	500	10,000	960	Extended press cure in lieu of postcure
491	K-83-1 K-83-2	3" x 1-5/8" dia cylinder	TP(H)-35-C	1/1 acetone	Spatula coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1
534	K-88-1 K-88-2 K-88-3 K-88-4	3" x 1-5/8" dia cylinder	9-35-C	1/0.5 acetone	Spatula coating	60	60 min at 160°F	20 min at 225°F	300	10,000	120	B-1
535a	K-89-1 K-89-2 K-89-3 K-89-4	3" x 1-5/8" dia cylinder	9-35-R	1/0.5 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	10,000	120	B-1
535b	K-89-5 K-89-6	3" x 1-5/8" dia cylinder	9-35-R	1/0.5 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	300	10,000	120	B-1

TABLE VIII (CONTINUED)

FABRICATION DETAILS — ROCKET NOZZLES

Data Sheet Number	Nozzle Number	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			+Postcure
						Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time, Minutes	
536	K-90-1 K-90-2	3" x 1-3/4" x 1-3/4" block	PY1-35-C	Used as received	Dip coating	10 min between dips	3 min at 200°F after every other dip	12 min at 200°F	300	1000	60	L-1
538	K-91-1 K-91-2	3" x 1-3/4" x 1-3/4" block	SC1-35-T25	Used as received	Prepared by AFML	—	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
540/552	K-92-1	3" x 1-3/4" x 1-3/4" block	SC1-35-WYB85	Used as received	Prepared by AFML	—	60 min at 160°F	40 min at 225°F	300	10,000	120	B-1
542/553	K-93-1	3" x 1-3/4" x 1-3/4" block	SC1-35-VYB70	Used as received	Prepared by AFML	—	60 min at 160°F	60 min at 225°F	300	10,000	120	B-1
542b	K-93-2	3" x 1-3/4" x 1-3/4" block	SC1-35-VYB70	Used as received	Prepared by AFML	—	60 min at 160°F	45 min at 225°F	300	10,000	120	B-1
546	K-95-1 K-95-2	3" x 1-3/4" x 1-3/4" block	DP25-35-T25	Used as received	Prepared by AFML	—	60 min at 160°F	30 min at 225°F	300	10,000	120	B-1
562	K-97-1 K-97-2	3" x 1-5/8" dia cylinder	9-FP-30-C	1/2 acetone	Spatula coating	60	45 min at 160°F	15 min at 225°F	300	10,000	120	B-1
563	K-100-1 K-100-2	3" x 1-5/8" dia cylinder	9-BBB-30-C	1/4 acetone	Dip coating	60	45 min at 160°F	—	300	10,000	120	B-1
566	K-101-1 K-101-2	3" x 1-5/8" dia cylinder	9-35-T40F	1/3 acetone	Spatula coating	60	45 min at 160°F	25 min at 225°F	300	10,000	120	B-1
567	K-102-1	3" x 1-5/8" dia cylinder	PP412B-35-C	Used as received	Brush coating	20	20 min at 160°F	—	400	10,000	120	I-3
571	K-104-1 K-104-2	3" x 1-5/8" dia cylinder	9-35-C	1/2.2 acetone	Brush coating	60	45 min at 160°F	25 min at 225°F	300	10,000	120	B-1

+The following postcure schedules were used

B-1 18 hrs at 275°F, 72 hrs from 275° to 400°F, 4 hrs at 400°F, 7 hrs cooling to below 200°F.

B-7 24 hrs at each of the following temperatures 375°, 435°, 475°, and 575°F, 4 hrs at 700°F, 6 hrs between temperatures. Cooled to below 200°F. Parts were postcured in argon.

B-8 16 hrs at 275°F, 72 hrs from 275° to 400°F, 6 hrs from 400° to 450°F, 4 hrs at 450°F, 6 hrs from 450° to 500°F, 12 hrs at 500°F. Cooled to below 200°F.

I-3 18 hrs at 275°F, 108 hrs from 275° to 550°F, 6 hrs at 550°F, 7 hrs cooling to below 200°F. Parts were postcured in argon.

I-2 3 hrs each at 150°, 200°, 250°, 300°, 350°F, 6 hrs at 400°F. Cooled to below 200°F before removing.

L-1 165 hrs from 275° to 600°F, 27 hrs at 600°F, 7 hrs cooling to below 200°F.

TABLE IX

FABRICATION DETAILS—MISCELLANEOUS TYPES OF SPECIMENS

Data Sheet Number	Type of Specimen	Dimensions of Original Laminate or Molding	Material Code	Ratio of Resin to Solvent	Type of Impregnation	Drying Conditions		B-Staging Conditions	Molding Conditions			+Postcure
						Air Dry, Minutes	Oven Dry		Temp., °F	Pressure, PSI	Time, Minutes	
462	Laminated square	2-1/2" x 2-1/4" x 5/8" laminate	F171-40-C	1/2 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	450	1500	120	B-7
463	Laminated square	4-1/2" x 2-1/2" x 5/8" laminate	F172-40-C	1/1 acetone	Spatula coating	60	60 min at 160°F	25 min at 225°F	350	1500	120	B-8
525-1 525-2	Cylinder	2" x 1" dia cylinder	T-100	Not applicable	Not applicable	Not applicable	Not applicable	Not applicable	700 700	5000 5000	15 420	None
527	Laminate	7" x 7" x 1/4"	PP412-40-AQ570	Used as received	Spatula coating	30	25 min at 160°F	Vacuum dried at room temp for 18 hrs	400	300	120	I-3
529-1 529-2	Cylinder	2" x 1" dia cylinder	PP412-40-AQ570	Used as received	Dip coating	30	25 min at 160°F	Vacuum dried at room temp for 18 hrs	400	10,000	120	I-3
554	Molding	1-7/8" x 1-1/2" x 1-1/4" block	PP413-35-AQ570	Used as received	Spatula coating	16 (hrs)	–	15 min at 160°F[1]	400	5000	120	I-3
572a-1	TGA	–	Polyphenylene sulfide (Phillips)	Not applicable	Not applicable				650	3300	120	I-3
572a-2			Abchar L913	Not applicable	Not applicable	Vacuum dried to a powder			400	500	120	I-3
572a-3			Abchar 412B	Not applicable	Not applicable	Vacuum dried to a powder			400	500	120	I-3
572b-4	TGA	–	91LD	Not applicable	Not applicable	91LD resin was vacuum dried to a powder before combining with the required fillers			300	500	120	B-1
572b-5			91LD & poly-(perfluoro-phenylene)									
572b-6			91LD & BBB polymer									

[1] Plies were vacuum dried at room temperature for two hours after B-Staging.

+The following postcure schedules were used:

B-1 18 hrs at 275°F, 72 hrs from 275°F to 400°F, 4 hrs at 400°F, 7 hrs cooling to below 200°F.

B-7 24 hrs at each of the following temperatures 375°, 435°, 475°, and 575°F, 4 hrs at 700°F, 6 hrs between temperatures. Cooled to below 200°F. Parts were postcured in argon.

B-8 16 hrs at 275°F, 72 hrs from 275° to 400°F, 6 hrs from 400° to 450°F, 4 hrs at 450°F, 6 hrs from 450° to 500°F, 12 hrs at 500°F, cooled to below 200°F.

I-3 12 hrs at 500°F. Cooled to below 200°F. 18 hrs at 275°F, 108 hrs from 275° to 550°F, 6 hrs at 550°F, 7 hrs cooling to below 200°F. Specimens were postcured in argon.

TABLE X

MATERIAL SOURCES

Trade Name or Designation	Type of Material	Source
91LD (phenolic)	Resin	American Reinforced Sales
Abchar 412 (polyphenylene)*	Resin	Hughes Aircraft
Abchar 412B (polyphenylene)*	Resin	Hughes Aircraft
Abchar 413 (polyphenylene)*	Resin	Hughes Aircraft
Abchar 700 (polyphenylene)*	Resinous filler	Hughes Aircraft
Abchar H913 (polyphenylene)*	Resin	Hughes Aircraft
Abchar L913 (polyphenylene)*	Resin	Hughes Aircraft
Abchar H1013 (polyphenylene)*	Resin	Hughes Aircraft
Abchar L1112 (polyphenylene)*	Resin	Hughes Aircraft
Astroquartz 570 (high silica content fabric)	Reinforcement	AFML (J. P. Stevens)
Astrosil 84 (high silica content fabric)	Reinforcement	AFML (J. P. Stevens)
Astrosil 11341B (high silica content fabric)	Reinforcement	AFML (J. P. Stevens)
Bisbenzimidazobenzophenanthroline	Resinous filler	AFML
Boron nitride fibers	Reinforcement	AFML
Carbon cloth CCA-1	Reinforcement	HITCO
Carbon cloth CCA-1 (low alkalinity-SS1641)	Reinforcement	HITCO
Carbon yarn VYB70 1/2	Reinforcement	AFML (Union Carbide)
Chrome-P (chrome-phenolic)	Resin	Thermo Resist
DEN 438 (epoxy novolac)	Resin	Dow Chemical
DP4-31 (phenyl aldehyde)	Resin	Ironsides Resins
DP25-10 (phenyl aldehyde)	Resin	Ironsides Resins
F170 (polyimide)	Resin	Coast Manufacturing and Supply
F171 (polyarylene-phenolic)	Resin	Coast Manufacturing and Supply

TABLE X (CONTINUED)

MATERIAL SOURCES

Trade Name or Designation	Type of Material	Source
F172 (polyphenylene-phenolic)	Resin	Coast Manufacturing and Supply
Graphite cloth G1550, uncoated	Reinforcement	HITCO
Graphite yarn WYB85 1/2	Reinforcement	AFML (Union Carbide)
PBI-carborane	Resin	AFML
p-Phenylphenol phenol formaldehyde	Resin	Hughes Aircraft
Poly(α, α'-diphenyl-m-xylylidene)	Resin	AFML
Poly(α, α'-diphenyl-p-xylylidene)	Resin	AFML
Polyaminoborane	Resinous filler	AFML
Polyimidazopyrrolone (Pyrrone)	Resin	Narmco
Poly(perfluorophenylene)	Resinous filler	AFML
Polyphenylene sulfide	Resin	Phillips Petroleum
Polyphenylene sulfide QX4375.4	Resin	Dow Chemical
Quartz yarn	Reinforcement	AFML
Refrasil C100-48 (high silica content fabric)	Reinforcement	HITCO
SC1008 (phenolic)	Resin	Monsanto
Silicon carbide whiskers	Reinforcement	AFML
Skybond 703 (polyimide)	Resin	Monsanto
Teflon 30 (polytetrafluoroethylene)	Resin	AFML
Thornel 25 graphite fibers	Reinforcement	AFML (Union Carbide)
Thornel 40 graphite fabric	Reinforcement	AFML (Union Carbide)
Tungsten-P (high tungsten content phenolic)	Resin	Thermo Resist

*Information on these resins is given on pages 36, 37, 38, 39, and 40

TABLE XI

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
—	Pellet	9-3-R	ASD-TDR-63-568,I
—	Pellet	9-4-R	ASD-TDR-63-568,I
—	Pellet	9-3-C	ASD-TDR-63-568,I
—	Pellet	D-3-R	ASD-TDR-63-568,I
—	Pellet	D-4-R	ASD-TDR-63-568,I
—	Pellet	D-3-C	ASD-TDR-63-568,I
—	Pellet	D-4-C	ASD-TDR-63-568,I
—	Pellet	W-3-R	ASD-TDR-63-568,I
—	Pellet	B-3-R	ASD-TDR-63-568,I
—	Pellet	B-4-R	ASD-TDR-63-568,I
—	Pellet	B-3-C	ASD-TDR-63-568,I
—	Pellet	N-4-R	ASD-TDR-63-568,I
—	Pellet	PNPII-2-R	ASD-TDR-63-568,I
—	Pellet	PNPII-4-C	ASD-TDR-63-568,I
S-45	Nozzle	9ILD-Carbon Cloth CCA-1	ASD-TDR-63-568,I
S-46	Nozzle	9ILD-WCB Graphite Cloth and Refrasil Cloth C100-48	ASD-TDR-63-568,I
S-47	Nozzle	9ILD-WCB Graphite Cloth and Refrasil Cloth C100-48	ASD-TDR-63-568,I
S-48	Nozzle	9ILD-Refrasil Cloth C100-48	ASD-TDR-63-568,I
S-49	Nozzle	9ILD-WCB Graphite	ASD-TDR-63-568,I
S-50	Nozzle	ATJ-Graphite Block	ASD-TDR-63-568,I
S-51	Nozzle	Polybenzimidazole-Refrasil C100-48	ASD-TDR-63-568,I
1	Pellet	9-4-C	ASD-TDR-63-568,I
2	Pellet	W-3-C	ASD-TDR-63-568,I
3	Pellet	B-4-C	ASD-TDR-63-568,I
41	Pellet	P-3-R	ASD-TDR-63-568,I
43	Pellet	D-4-TR	ASD-TDR-63-568,I
44	Pellet	D-3-TR	ASD-TDR-63-568,I
48	Pellet	D-3-TC	ASD-TDR-63-568,I
50	Pellet	D-4-TC	ASD-TDR-63-568,I
51	Pellet	D-4-BR	ASD-TDR-63-568,I
52	Pellet	D-3-BR	ASD-TDR-63-568,I
52	Pellet	9-3-ZA	ASD-TDR-63-568,I
53	Pellet	D-4-BC	ASD-TDR-63-568,I
53	Pellet	9-4-ZA	ASD-TDR-63-568,I
56	Pellet	PN-4-P	ASD-TDR-63-568,I
57	Pellet	PN-4-R	ASD-TDR-63-568,I
58	Pellet	PN-3-R	ASD-TDR-63-568,I
62	Pellet	PE-3-R	ASD-TDR-63-568,I
65	Pellet	PE-3-C	ASD-TDR-63-568,I
66	Pellet	PE-4-C	ASD-TDR-63-568,I
67	Pellet	PN-4-C	ASD-TDR-63-568,I
68	Pellet	PN-4-C	ASD-TDR-63-568,I
69	Pellet	PE-4-R	ASD-TDR-63-568,I
71	Pellet	PS-3-R	ASD-TDR-63-568,I
72	Pellet	PS-4-R	ASD-TDR-63-568,I
73	Pellet	PS-3-C	ASD-TDR-63-568,I
74	Pellet	PS-4-C	ASD-TDR-63-568,I
75	Pellet	PLP-3-R	ASD-TDR-63-568,I
76	Pellet	PP-3-R	ASD-TDR-63-568,I
77	Pellet	PP-4-R	ASD-TDR-63-568,I
79	Pellet	PP-4-C	ASD-TDR-63-568,I
101	Pellet	PN-4 R	ASD-TDR-63-568,I
102	Pellet	PN-3-R	ASD-TDR-63-568,I
103	Pellet	PN-4-R	ASD-TDR-63-568,I
105	Pellet	PN-3-C	ASD-TDR-63-568,I
106	Pellet	N-4-C	ASD-TDR-63-568,I
111	Pellet	PNPII-4-R	ASD-TDR-63-568,I
112	Pellet	PNPII-4-C	ASD-TDR-63-568,I
114	Pellet	9-3-H	ASD-TDR-63-568,I
115	Pellet	D-3-H	ASD-TDR-63-568,I
116	Pellet	D-4-H	ASD-TDR-63-568,I
117	Pellet	9-3-HC	ASD-TDR-63-568,I
118	Pellet	9-3-HR	ASD-TDR-63-568,I
119	Pellet	9-3-O	ASD-TDR-63-568,I
120	Pellet	R-3-H	ASD-TDR-63-568,I
121	Pellet	9-4-ZA	ASD-TDR-63-568,I
122	Pellet	9-3-ZE	ASD-TDR-63-568,I
123	Pellet	9-3-ZA	ASD-TDR-63-568,I
123	Pellet	9-3-ZE	ASD-TDR-63-568,I
124	Pellet	D-3-ZA	ASD-TDR-63-568,I

TABLE XI (CONTINUED)

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
127	Pellet	9-3-ZC	ASD-TDR-63-568,I
127	Pellet	9-Co-35R	ML-TDR-64-222
158	Pellet	9-V-35R	ML-TDR-64-222
159	Pellet	9-Z-35R	ML-TDR-64-222
160	Pellet	9-Z-35C	ML-TDR-64-222
162	Nozzle	N9-4-R	ML-TDR-64-222
163	Nozzle	N9-4-RD	ML-TDR-64-222
164	Nozzle	N9-4-CD	ML-TDR-64-222
168/247	Pellet	9-35VFA(PG) with 9-35-C Backing	ML-TDR-64-222
169	Laminate	DN-30-G	ML-TDR-64-222
170	Laminate	PP-30-G-1	ML-TDR-64-222
171	Laminate	PP-30-G-2	ML-TDR-64-222
172	Pellet	9-35-WFA(PG)	ML-TDR-64-222
173	Nozzle	N-35-R	AFML-TR-66-75,I
174	Pellet	9-35-VFA	ML-TDR-64-222
175	Pellet	9-35-VFA(PG)	ML-TDR-64-222
176	Pellet	9-35-WFA	ML-TDR-64-222
179	Pellet	R-35-HC	ML-TDR-64-222
184	Pellet	9-35-GU	ML-TDR-64-222
186	Pellet	9-35-GC	ML-TDR-64-222
187	Pellet	9-35-HC	ML-TDR-64-222
191	Pellet	9-35-ZC	ML-TDR-64-222
196	Pellet	D-35-ZF	ML-TDR-64-222
197	Laminate	9-35-C	ML-TDR-64-222
199	Nozzle	D-35-7-11	ML-TDR-64-222
200	Nozzle	9-40-IRD	ML-TDR-64-222
202	Pellet	9-35-C	ML-TDR-64-222
203	Pellet	9-35-GU	ML-TDR-64-222
204	Pellet	9-35-C(PG)	ML-TDR-64-222
205	Laminate	9-35-R	ML-TDR-64-222
206	Laminate	9-35-G	ML-TDR-64-222
207	Pellet	D-45-ZF	ML-TDR-64-222
208	Pellet	9-35-R	ML-TDR-64-222
213	Pellet	DN-35-C	ML-TDR-64-222
216	Pellet	D-35-7	ML-TDR-64-222
217	Nozzle	9-40-GC	ML-TDR-64-222
218	Pellet	SE-35-C	ML-TDR-64-222
220	Pellet	SE-35-R	ML-TDR-64-222
222	Pellet	DN-45-C	ML-TDR-64-222
223	Pellet	DN-45-R	ML-TDR-64-222
224	Pellet	DN-35-R	ML-TDR-64-222
225	Pellet	SE-45-C	ML-TDR-64-222
226	Pellet	SE-45-R	ML-TDR-64-222
227	Pellet	PP-35-R	ML-TDR-64-222
228	Pellet	PP-45-R	ML-TDR-64-222
229	Nozzle	9-40-C	ML-TDR-64-222
231	Nozzle	9-40-R	ML-TDR-64-222
232a&b	Nozzle	9-40-CR	ML-TDR-64-222
233	Pellet	PP-35-C	ML-TDR-64-222
234	Pellet	PP-45-C	ML-TDR-64-222
235	Pellet	9-45-PBIF	AFML-TR-66-75,I
236	Pellet	9-35-PBIF	AFML-TR-66-75,I
237	Pellet	DPO-35-C	ML-TDR-64-222
238	Pellet	DPO-35-C	ML-TDR-64-222
239	Pellet	DPO-35-R	ML-TDR-64-222
240	Pellet	DPO-45-R	ML-TDR-64-222
241	Nozzle	9-TB-40-C	ML-TDR-64-222
242	Nozzle	9-MB-40-C	ML-TDR-64-222
243	Nozzle	9-BC-40-C	ML-TDR-64-222
244	Nozzle	9-40-G	ML-TDR-64-222
245	Laminate	DN-30-G-2	ML-TDR-64-222
246/247	Pellet	9-35-PGW with 9-35-C Backing	ML-TDR-64-222
247	See 168 and 246	9-35-C	ML-TDR-64-222
248	Pellet	9-35-CTC	ML-TDR-64-222
249	Pellet	9-35-GTC	ML-TDR-64-222
252	Pellet	DN-45-C	ML-TDR-64-222
253	Pellet	DN-35-C	ML-TDR-64-222
254	Pellet	HB-45-C	ML-TDR-64-222
255	Pellet	HB-35-C	AFML-TR-65-94
256	Nozzle	9-40-CCZ	ML-TDR-64-222

TABLE XI (CONTINUED)

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
257	Nozzle	9-40-GCZ	ML-TDR-64-222
258	Pellet	9-35-CCZ	ML-TDR-64-222
259	Pellet	9-35-GCZ	ML-TDR-64-222
260	Nozzle	9-40-R	AFML-TR-65-94
261	Nozzle	9-40-C	AFML-TR-65-94
262	Pellet	9-35-GCTi	AFML-TR-65-94
263	Pellet	9-35-GCHf	AFML-TR-65-94
264	Nozzle	9-40-GCHf	AFML-TR-65-94
265	Nozzle	9-40-GCTi	AFML-TR-65-94
266	Pellet	PS-45-C	AFML-TR-65-94
268	Pellet	PS-35-C	AFML-TR-65-94
269	Hot Gas Flow	9-40-R	AFML-TR-65-94
270	Pellet	PDB-45-C	AFML-TR-65-94
271	Hot Gas Flow	9-40-C	AFML-TR-65-94
272	Pellet	I8-40-C	AFML-TR-65-94
273	Pellet	I8-40-R	AFML-TR-65-94
274	Nozzle	I8-40-C	AFML-TR-65-94
275	Nozzle	I8-40-R	AFML-TR-65-94
276	Hot Gas Flow	I8-40-C	AFML-TR-65-94
277	Hot Gas Flow	I8-40-R	AFML-TR-65-94
278	Hot Gas Flow	I8-40-C	AFML-TR-65-94
279	Hot Gas Flow	I8-40-R	AFML-TR-65-94
280	Pellet	SG7-35-R	AFML-TR-65-94
281	Pellet	PBB-45-C	AFML-TR-65-94
282	Pellet	D-35-WFA	AFML-TR-65-94
283	Pellet	D-35-GU	AFML-TR-65-94
285	Hot Gas Flow	9-40-C	AFML-TR-65-94
286	Hot Gas Flow	9-40-R	AFML-TR-65-94
287	Hot Gas Flow	9-40-R	AFML-TR-65-94
288	Hot Gas Flow	9-40-C	AFML-TR-65-94
289	Nozzle	9-40-GC(2.5)	AFML-TR-65-94
290	Pellet	PPP-35-C	AFML-TR-65-94
291	Pellet	DN-35-C	AFML-TR-65-94
292	Laminate	PPP-35-G	AFML-TR-65-94
293	Laminate	DN-35-G	AFML-TR-65-94
295	Hot Gas Flow	DN-40-C	AFML-TR-65-94
297	Pellet	SG7-45-R	AFML-TR-65-94
298	Hot Gas Flow	9-40-C	AFML-TR-65-94
299	Pellet	SG7-35-C	AFML-TR-65-94
300	Molding	9-35-SGF	AFML-TR-65-94
301	Pellet	SG7-45-C	AFML-TR-65-94
302	Pellet	DN-35-C	AFML-TR-65-94
303	Nozzle	DN-40-C	AFML-TR-65-94
304	Nozzle	DN-40-R	AFML-TR-65-94
305	Molding	9-55-HGF	AFML-TR-65-94
306	Pellet	PP-45-C	AFML-TR-65-94
307	Pellet	PP-35-C	AFML-TR-65-94
309	Nozzle	D-40-C	AFML-TR-65-94
310	Laminate	PP-35-G	AFML-TR-65-94
311	Nozzle	PP-40-C	AFML-TR-65-94
312	Pellet	9-35-C	AFML-TR-65-94
313	Nozzle	PP-40-R	AFML-TR-65-94
315	Pellet	9-35-GC	AFML-TR-65-94
316	Hot Gas Flow	PP-40-C	AFML-TR-65-94
317	Hot Gas Flow	PP-40-R	AFML-TR-65-94
318	Pellet	9-35-GC(2.5)	AFML-TR-65-94
324	Hot Gas Flow	DN-40-R	AFML-TR-65-94
325	Nozzle	PPP-40-C	AFML-TR-65-94
326	Nozzle	PPP-40-R	AFML-TR-65-94
327	Hot Gas Flow	PPP-40-R	AFML-TR-65-94
328	Hot Gas Flow	PPP-40-C	AFML-TR-65-94
330	Pellet	9-35-GCB	AFML-TR-66-75, I
331	Pellet	9-35-SW	AFML-TR-66-75, I
333	Pellet	9-35-SCW	AFML-TR-66-75, I
334	Pellet	9-35-SC70	AFML-TR-66-75, I
335	Pellet	9-35-T70	AFML-TR-66-75, I
336	Pellet	9-35-RF	AFML-TR-66-75, I
337	Nozzle	9-35-RF	AFML-TR-66-75, I
338	Pellet	9-35-RSF	AFML-TR-66-75, I
339	Nozzle	9-40-RSF	AFML-TR-66-75, I

TABLE XI (CONTINUED)

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
340	Nozzle	N-35-C (SY)	AFML-TR-66-75, I
341	Laminated Square	N-35-C (SY)	AFML-TR-66-75, I
342	Pellet	N-35-C (SY)	AFML-TR-66-75, I
343	Nozzle	N-35-C (S)	AFML-TR-66-75, I
344	Laminated Square	N-35-C (S)	AFML-TR-66-75, I
345	Pellet	N-35-C (S)	AFML-TR-66-75, I
346	Nozzle	N-35-C(D)	AFML-TR-66-75, I
347	Laminated Square	N-35-C(D)	AFML-TR-66-75, I
348	Pellet	N-35-C(D)	AFML-TR-66-75, I
349	Nozzle	N-35-C(SE)	AFML-TR-66-75, I
350	Laminated Square	N-35-C(SE)	AFML-TR-66-75, I
351	Pellet	N-35-C(SE)	AFML-TR-66-75, I
352	Pellet	PS-35-RCB	AFML-TR-66-75, I
353	Hot Gas Flow	9-40-C	AFML-TR-66-75, I
354	Hot Gas Flow	9-40-R	AFML-TR-66-75, I
356	Nozzle	N151-40-G	AFML-TR-66-75, I
357	Hot Gas Flow	9-40-C	AFML-TR-66-75, I
358	Hot Gas Flow	9-40-R	AFML-TR-66-75, I
359	Nozzle	9-40-CLA	AFML-TR-66-75, I
360	Pellet	9-35-CLA	AFML-TR-66-75, I
361	Pellet	PP413-35-C	AFML-TR-66-75, II
362	Pellet	PP413-35-R	AFML-TR-66-75, II
363	Laminate	PP413-35-G	AFML-TR-66-75, II
364	Nozzle	N151-40-R	AFML-TR-66-75, I
368	Laminate	PP413-35-G	AFML-TR-66-75, I
369	Pellet	PP413-35-C	AFML-TR-66-75, I
370	Pellet	9PP-45-C	AFML-TR-66-75, I
372	Nozzle	PP(413)-40-C	AFML-TR-66-75, I
373	Nozzle	CP-40-R	AFML-TR-66-75, I
374	Pellet	CP-35-R	AFML-TR-66-75, I
375	Pellet	CP-35-C	AFML-TR-66-75, I
376	Pellet	9-35-GCB(2. 5)	AFML-TR-66-75, I
377/394	Pellet	N151-35-C	AFML-TR-66-75, II
378/394	Hot Gas Flow	N151-35-C	AFML-TR-66-75, II
379/394	Laminated Square	N151-35-C	AFML-TR-66-75, II
380/382	Pellet	N151-35-R	AFML-TR-66-75, II
381/382	Laminated Square	N151-35-R	AFML-TR-66-75, II
383	Hot Gas Flow	9-40-C	AFML-TR-66-75, I
384	Nozzle	9-40-CSF	AFML-TR-66-75, I
385	Pellet	9-35-CSF	AFML-TR-66-75, I
386	Nozzle	D-40-R	AFML-TR-66-75, I
387	Pellet	PNPII-40-A	AFML-TR-66-75, I
388	Pellet	PNPII-40-R	AFML-TR-66-75, I
389	Pellet	9-35-RSF	AFML-TR-66-75, I
390	Pellet	9-35-FSF(PC)	AFML-TR-66-75, I
391	Pellet	9-35-14	AFML-TR-66-75, II
392	Nozzle	N151-40-C	AFML-TR-66-75, I
393	Nozzle	N151-40-R	AFML-TR-66-75, I
395a	Hot Gas Flow	9-40-C	AFML-TR-66-75, I
395b	Hot Gas Flow	9-40-C	AFML-TR-66-75, I
396	Pellet	D-30-BF	AFML-TR-66-75, I
397a	Nozzle	GR1-40-C	AFML-TR-66-75, II
397b	Pellet	GR1-40-C	AFML-TR-66-75, II
399a	Nozzle	9-40-C	AFML-TR-66-75, I
399b	Nozzle	9-40-C	AFML-TR-66-75, I
400	Nozzle	9-40-GU	AFML-TR-66-75, I
401	Nozzle	9-40-R	AFML-TR-66-75, I
402a	Nozzle	D-30-C	AFML-TR-66-75, I
402b	Cylindrical Blank	D-30-C	AFML-TR-66-75, I
402c	Nozzle	D-30-C	AFML-TR-66-75, I
403a	Nozzle	SG7-40-C	AFML-TR-66-75, I
403b	Nozzle	SG7-40-C	AFML-TR-66-75, II
405	Nozzle	D-30-R	AFML-TR-66-75, II
406	Nozzle	PP(412)-40-C	AFML-TR-66-75, I
407	Nozzle	F170-40-C	AFML-TR-66-75, III
408	Nozzle	F171-40-C	AFML-TR-66-75, III
409	Nozzle	F172-40-C	AFML-TR-66-75, III
410	Nozzle	PH9-40-C	AFML-TR-66-75, I
413	Pellet	D-35-BN	AFML-TR-66-75, I
414	Supersonic Pipe	N151-35-C	AFML-TR-66-75, I

TABLE XI (CONTINUED)

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
415	Supersonic Pipe	PP(412)-35-GU	AFML-TR-66-75, I
416	Supersonic Pipe	PPP-35-GU	AFML-TR-66-75, I
417	Supersonic Pipe	DN-35-GU	AFML-TR-66-75, I
418	Supersonic Pipe	9-35-GU	AFML-TR-66-75, I
419	Supersonic Pipe	9-35-GU	AFML-TR-66-75, I
420	Supersonic Pipe	SG7-35-GU	AFML-TR-66-75, I
421	Supersonic Pipe	9-35-GC	AFML-TR-66-75, I
422	Pellet	9-35-QF	AFML-TR-66-75, I
423	Nozzle	PPF-40-C	AFML-TR-66-75, I
424a	Nozzle	PPF-40-R	AFML-TR-66-75, I
424b	Nozzle	PPF-40-R	AFML-TR-66-75, I
425	Supersonic Pipe	9-35-C	AFML-TR-66-75, I
426	Pellet	PH9-35-R	AFML-TR-66-75, II
427	Pellet	PH9-35-C	AFML-TR-66-75, II
428	Pellet	PP500-35-C	AFML-TR-66-75, II
429	Pellet	PP500-35-R	AFML-TR-66-75, II
431	Pellet	PP413-PP600-30-C	AFML-TR-66-75, II
432	Nozzle	PP413-PP600-30-C	AFML-TR-66-75, II
433	Nozzle	PP413-PP600-30-R	AFML-TR-66-75, II
433b	Nozzle	PP413-PP600-30-R	AFML-TR-66-75, II
440	Pellet	9-28-SCWC	AFML-TR-
442/434	Hot Gas Flow	DDPO-35-GU	AFML-TR-66-75, II
443/434	Laminated Square	DDPO-35-GU	AFML-TR-66-75, II
444/434	Pellet	DDPO-35-GU	AFML-TR-66-75, II
445/435	Hot Gas Flow	SG7-35-GU	AFML-TR-66-75, II
446/435	Laminated Square	SG7-35-GU	AFML-TR-66-75, II
447/435	Pellet	SG7-35-GU	AFML-TR-66-75, II
448/436	Hot Gas Flow	CP-35-GU	AFML-TR-66-75, II
449/436	Laminated Square	CP-35-GU	AFML-TR-66-75, II
450/436	Pellet	CP-35-GU	AFML-TR-66-75, II
451/437	Hot Gas Flow	PPP-35-GU	AFML-TR-66-75, I
452/437	Laminated Square	PPP-35-GU	AFML-TR-66-75, I
453/437	Pellet	PPP-35-GU	AFML-TR-66-75, I
454/438	Hot Gas Flow	DNB-35-GU	AFML-TR-66-75, II
455/438	Laminated Square	DNB-35-GU	AFML-TR-66-75, II
456/438	Pellet	DNB-35-GU	AFML-TR-66-75, II
457/441	Hot Gas Flow	9-35-GU	AFML-TR-66-75, I
458/441	Laminated Square	9-35-GU	AFML-TR-66-75, I
459/441	Pellet	9-35-GU	AFML-TR-66-75, I
460a	Pellet	9-28-BF	AFML-TR-66-75, II
460b	Pellet	9-28-BF	AFML-TR-66-75, II
462	Laminated Square	F171-40-C	AFML-TR-66-75, III
463	Laminated Square	F172-40-C	AFML-TR-66-75, III
465	Pellet	D-35-BNF	AFML-TR-66-75, II
466a	Nozzle	D-28-BF	AFML-TR-66-75, II
466b	Nozzle	D-28-BF	AFML-TR-66-75, II
467	Nozzle	CP-35-C	AFML-TR-66-75, III
468-1a	Nozzle	PPS(5.4)NS-35-C	AFML-TR-66-75, III
468-1b	Nozzle	PPS(5.4)PT-35-C	AFML-TR-66-75, III
469	Nozzle	PPS(5.4)PT-35-R	AFML-TR-66-75, III
473-1a	Pellet	PPS(5.4)NS-35-C	AFML-TR-66-75, III
473-1b	Pellet	PPS(5.4)PT-35-C	AFML-TR-66-75, III
473-2a	Pellet	PPS(5.4)NS-35-C	AFML-TR-66-75, III
473-2b	Pellet	PPS(5.4)PT-35-C	AFML-TR-66-75, III
477	Pellet	D-35-R	AFML-TR-66-75, II
478	Pellet	D-PAB-25-R	AFML-TR-66-75, III
479	Pellet	D-35-AS84	AFML-TR-66-75, III
480	Pellet	D-35-AS41B	AFML-TR-66-75, III
481	Pellet	9-FP-30-GU	AFML-TR-66-75, III
482	Pellet	PBIC-35-R	AFML-TR-66-75, III
483 a&b	Pellet	9-PBIC-30-GU	AFML-TR-66-75, III
484	Pellet	9-BBB-30-GU	AFML-TR-66-75, III
485	Pellet	DPPX-35-GU	AFML-TR-66-75, III
486	Pellet	DPMX-35-GU	AFML-TR-66-75, III
489	Nozzle	9-35-AQ570	AFML-TR-66-75, II
490	Nozzle	9-35-AS41B	AFML-TR-66-75, II
491	Nozzle	TP(H)-35-C	AFML-TR-66-75, III
494/487	Hot Gas Flow	TP(H)-35-GU	AFML-TR-66-75, II
495/487	Laminated Square	TP(H)-35-GU	AFML-TR-66-75, II
496/487	Pellet	TP(H)-35-GU	AFML-TR-66-75, II

TABLE XI (CONTINUED)

CUMULATIVE INDEX OF SPECIMENS

Data Sheet Number	Type of Specimen	Material Code	Fabrication Data in Report Number
497/488	Hot Gas Flow	PPO-35-GU	AFML-TR-66-75, II
498/488	Laminated Square	PPO-35-GU	AFML-TR-66-75, II
499/488	Pellet	PPO-35-GU	AFML-TR-66-75, II
500	Hot Gas Flow	9-35-C	AFML-TR-66-75, II
501	Hot Gas Flow	170-35-C	AFML-TR-66-75, II
502	Hot Gas Flow	171-35-C	AFML-TR-66-75, II
503	Hot Gas Flow	172-35-C	AFML-TR-66-75, II
504	Hot Gas Flow	PP413-35-C	AFML-TR-66-75, II
505	Hot Gas Flow	DP4-35-C	AFML-TR-66-75, II
509	Nozzle	9-PP700-30-C	AFML-TR-66-75, II
510	Nozzle	PPP-PP700-30-C	AFML-TR-66-75, II
511	Nozzle	D-PP700-30-C	AFML-TR-66-75, II
512/506	Pellet	9-PP700-30-C	AFML-TR-66-75, II
513/507	Pellet	PPP-PP700-30-C	AFML-TR-66-75, II
514/508	Pellet	D-PP700-30-C	AFML-TR-66-75, II
515/506	Hot Gas Flow	9-PP700-30-C	AFML-TR-66-75, II
516/507	Hot Gas Flow	PPP-PP700-30-C	AFML-TR-66-75, II
517/508	Hot Gas Flow	D-PP700-30-C	AFML-TR-66-75, II
518	Pellet	PP412B-35-C	AFML-TR-66-75, III
520	Pellet	PP413-PP700-30-C	AFML-TR-66-75, III
521	Pellet	PP413-35-CLA	AFML-TR-66-75, III
522	Pellet	PP412B-PP700-30-C	AFML-TR-66-75, III
524	Pellet	T-BNF	AFML-TR-66-75, III
525	Cylinder	T-100	AFML-TR-66-75, III
526	Pellet	T-100	AFML-TR-66-75, III
527	Laminate	PP412-40-AQ570	AFML-TR-66-75, III
528	Laminate	PP412-40-AQ570	AFML-TR-66-75, II
529	Cylinder	PP412-40-AQ570	AFML-TR-66-75, III
530a, b&c	Pellet	PPL913-35-C	AFML-TR-66-75, III
531	Pellet	PPL1112-35-C	AFML-TR-66-75, III
532a, b&c	Pellet	PPH913-35-C	AFML-TR-66-75, III
533a, b&c	Pellet	PPH1013-35-C	AFML-TR-66-75, III
534	Nozzle	9-35-C	AFML-TR-66-75, III
535a&b	Nozzle	9-35-R	AFML-TR-66-75, III
536	Nozzle	PY1-35-C	AFML-TR-66-75, III
537	Pellet	PY1-35-C	AFML-TR-66-75, III
538	Nozzle	SC1-35-T25	AFML-TR-66-75, III
539 a&b	Pellet	SC1-35-T25	AFML-TR-66-75, III
540/552	Nozzle	SC1-35-WYB85	AFML-TR-66-75, III
541/552	Pellet	SC1-35-WYB85	AFML-TR-66-75, III
542/553	Nozzle	SC1-35-VYB70	AFML-TR-66-75, III
542b	Nozzle	SC1-35-VYB70	AFML-TR-66-75, III
543/553	Pellet	SC1-35-VYB70	AFML-TR-66-75, III
543b	Pellet	SC1-35-VYB70	AFML-TR-66-75, III
545	Pellet	SK703-35-T25	AFML-TR-66-75, III
546	Nozzle	DP25-35-T25	AFML-TR-66-75, III
547	Pellet	DP25-35-T25	AFML-TR-66-75, III
548	Pellet	SC1-35-QY	AFML-TR-66-75, III
549	Pellet	PPP-35-T25	AFML-TR-66-75, III
550	Pellet	PP413-35-T25	AFML-TR-66-75, III
551	Pellet	DP4-35-T25	AFML-TR-66-75, III
554	Molding	PP413-35-AQ570	AFML-TR-66-75, III
555	Pellet	PPSP-35-C	AFML-TR-66-75, III
556	Pellet	9-FP-30-C	AFML-TR-66-75, III
559	Pellet	9-BBB-30-C	AFML-TR-66-75, III
560	Pellet	9-35-T40F	AFML-TR-66-75, III
561	Pellet	PP412B-35-C	AFML-TR-66-75, III
562	Nozzle	9-FP-30-C	AFML-TR-66-75, III
565	Nozzle	9-BBB-30-C	AFML-TR-66-75, III
566	Nozzle	9-35-T40F	AFML-TR-66-75, III
567	Nozzle	PP412B-35-C	AFML-TR-66-75, III
571	Nozzle	9-35-C	AFML-TR-66-75, III
572 a&b	TGA		AFML-TR-66-75, III

TABLE XII

MATERIAL CODE SYMBOLS

KEY TO CODE SYMBOLS

Resin

Symbol	Material
9	91LD (phenolic)
170	F170 (polyimide)
171	F171 (polyarylene-phenolic)
172	F172 (polyphenylene-phenolic)
B	Phenyl-phenol-aldehyde (B4452-32)
CP	Chrome-P (chrome-phenolic)
D	DEN 438 (epoxy novolac)
DPPO	Diphenyl oxide QX-2682.1
DN	2,7 Dihydroxynaphthalene phenol formaldehyde
DNB	2,7 Dihydroxynaphthalene phenol formaldehyde (high barium content catalyst)
DP4	DP-4-31 (phenyl aldehyde)
DP25	DP-25-10 (phenyl aldehyde)
DPMX	α,α' Diphenyl-m-xylylidene
DPO	Doryl (diphenyl oxide)
DPPX	α,α' Diphenyl-p-xylylidene
F170	F170 (polyimide)
F171	F171 (polyarylene-phenolic)
F172	F172 (polyphenylene-phenolic)
GR1	GR-1 (organo phosphonitrilic)
HB	m - Hydroxybenzoic acid phenol formaldehyde
I8	I-8 (polyimide)
N	Imidite 1850 system (polybenzimidazole-phenol blocked)
N9	91LD (phenolic)
N151	Imidite 2803 system (AFR-151, polybenzimidazole-amine blocked)
P	ASD No. 091062A (polyester)
PBB	Polybenzodiazaboroline
PBIC	Polybenzimidazole-carborane
PDB	Polydioxaborole
PE	XHU - Unox 207 (phosphonitrilic-epoxide)
PH9	PH990 (organo phosphonitrilic)
PLP	Phenolic and polyphenyl
PN	XHU (phosphonitrilic)
PNPII	PNPII (carborane)
PP	Phospho-phenolic
PP	Polyphenylene
PP412	Abchar 412 (polyphenylene)
PP412B	Abchar 412B (polyphenylene)
PP413	Abchar 413 (polyphenylene)
PPF	2,2 bis (p-hydroxyphenyl) propane phenol formaldehyde
PPH913	Abchar H913 (high MW polyphenylene)
PPH1013	Abchar H1013 (high MW polyphenylene)
PPL913	Abchar L913 (low MW polyphenylene)
PPL1112	Abchar L1112 (low MW polyphenylene)
PPO	Polyphenylene oxide
PPP	p-Phenylphenol phenol formaldehyde
PPS(5.4)NS	Polyphenylene sulfide QX 4375.4 with sodium sulfide curing agent
PPS(5.4)PT	Polyphenylene sulfide QX 4375.4 with p-toluenesulfonic acid monohydrate and xylylene glycol as curing agents
PPSP	Polyphenylene sulfide (Phillips)
PS	Polyphenylene sulfide
PY1	Pyrrone (polyimidazopyrrolone)
R	Plyophen 23-057 (polyamide-phenolic)
S	R-7146 (silicone)
SC1	SC1008 (phenolic)
SE	QZ-8-0903 (silicone-epoxy)
SG7	Skybond 700 (formerly Skygard 700) (polyimide)
SK703	Skybond 703 (polyimide)
SY	Sylgard 182 (silicone)
T	Teflon 30 (tetrafluoroethylene)
TP(H)	Tungsten-P (high) (tungsten-phenolic)
W	A1-7 (polyamide-imide)

Reinforcement

Symbol	Material
7	Pyrolytic graphite filaments No. 7
11	Pyrolytic graphite filaments No. 11
14	Pyrolytic graphite fibers No. 14
A	Asbestos
AQ570	Astroquartz 570 (high silica content fabric)
AS41B	Astrosil 11341B (high silica content fabric)
AS84	Astrosil 84 (high silica content fabric)
BF	Boron fibers
BNF	Boron nitride fibers
C	Carbon cloth CCA-1
CCZ	Carbon cloth CCA-1 pyrolytic graphite-zirconia coated
CLA	Carbon cloth CCA-1, low alkalinity (SS1641)
C(PG)	Carbon cloth CCA-1-pyrolytic graphite coated
CSF	Carbon silica fabric
CTC	Carbon cloth CCA-1-tantalum carbide coated
FSF(PC)	Fused silica fabric-pyrolytic carbon coated
G	Glass cloth, style 181, A1100 finish
GC	Graphite cloth G1550-pyrolytic graphite coated-1 μ thick
GC(2.5)	Graphite cloth G1550-pyrolytic graphite coated-2.5 μ thick
GCB	Graphite cloth G1550-pyrolytic graphite-boron coated-1 μ thick
GCB(2.5)	Graphite cloth G1550-pyrolytic graphite-boron coated-2.5 μ thick
GC(Hf)	Graphite cloth G1550-pyrolytic graphite-hafnium coated
GC(Ti)	Graphite cloth G1550-pyrolytic graphite-titanium coated
GCZ	Graphite cloth G1550-pyrolytic graphite-zirconium coated
GTC	Graphite cloth G1550-tantalum carbide coated
GU	Graphite cloth G1550, uncoated
H	HT-1 fabric (polyamide)
HGF	Hollow glass fibers
IRD	Refrasil cloth 1554-48 (high silica content fabric)
O	Orlon fabric 10038/.2 (acrylic)
PBIF	Polybenzimidazole fibers
PGW	Pyrolytic graphite whiskers
QF	Quartz fabric 581
QY	Quartz yarn
R	Refrasil cloth C100-48 (high silica content fabric)
R-28	Refrasil cloth C100-28 (high silica content fabric)
RF	Rayon fabric
RSF	Rayon silica fabric
SC70	SC 70 silicon carbide fibers
SCW	Silicon carbide whiskers
SCW	Silicon carbide wool fibers
SGF	Solid glass fibers
SW	Sapphire wool fibers
T25	Thornel 25 graphite fibers
T40F	Thornel 40 graphite fabric
T70	T-70 fiber crystals
VFA	VFA carbon filaments
VFA(PG)	VFA carbon filaments-pyrolytic graphite coated
VYB70	Carbon yarn VYB70 1/2
WFA	WFA Graphite yarn
WFA(PG)	WFA graphite yarn-pyrolytic graphite coated
WYB85	Graphite yarn WYB 85 1/2
Z	Zirconia A fibers
ZA	Zirconia, Type A
ZC	Zirconia, Type C
ZE	Zirconia, Type E
ZF	Zirconia foil

Filler

Symbol	Material
B	Tetraboron carbide
BBB	Bisbenzimidazobenzophenanthroline resin
BC	Tetraboron carbide
CB	Carbon black 452-00156
Co	Cobalt Oxide
FP	Poly(perfluorophenylene) resin
MB	Molybdenum diboride
PAB	Polyaminoborane resin
PBIC	Polybenzimidazole-carborane resin
PP500	Abchar 500 (polyphenylene resin)
PP600	Abchar 600 (polyphenylene resin)
PP700	Abchar 700 (polyphenylene resin)
T	Titanium diboride
TB	Titanium diboride
V	Vanadium pentoxide

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13. ABSTRACT

Precise processing techniques were used in preparing new ablative plastics and composites. This research involved the use of novel heat resistant resins such as; a bisbenzimidazobenzophenanthroline; branched, crosslinked polyphenylenes; a chrome based metal phenolic; a PBI-carborane; phenyl aldehydes; poly (α, α' – diphenyl-xylylidines); a polyaminoborane; a polyimidazopyrrolone; polyimides; polyphenylene sulfides; a poly(perfluorophenylene); and a tungsten based metal organic phenolic.

Novel materials used as reinforcements included boron nitride fibers; a high modulus carbon yarn; high modulus graphite fabric and yarns; and silicon carbide whiskers.

Resin impregnation techniques used in preparing research specimens included spatula or brush coating, dip coating, soaking, and dry powder layup.

The following research specimens of controlled composition were prepared and submitted to the Air Force Materials Laboratory for hyperthermal evaluation: pellet specimens, 3/4-inch diameter by 1/2-inch long; rocket nozzle assemblies; cylinders, 1 inch diameter by 2 inches long; laminate, 7 x 7 x 1/4 inch; laminated squares, 2 x 2 x 1/2 inch.

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